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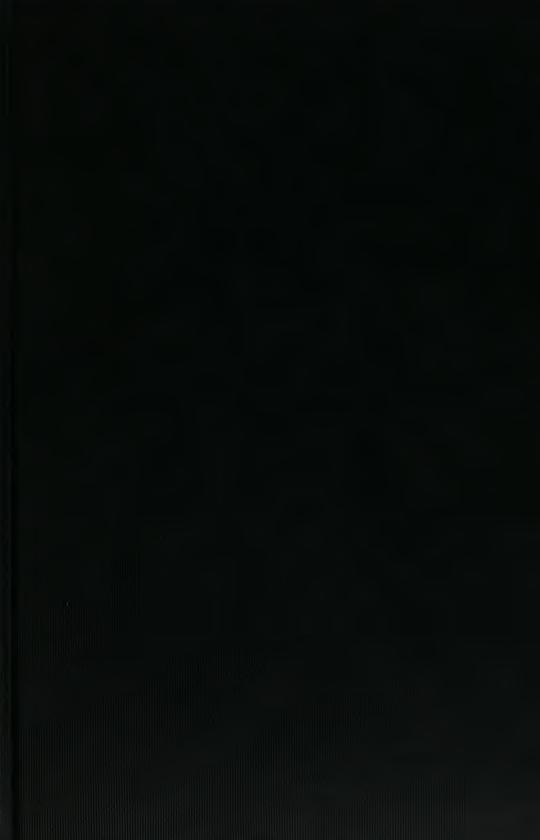
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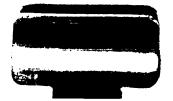


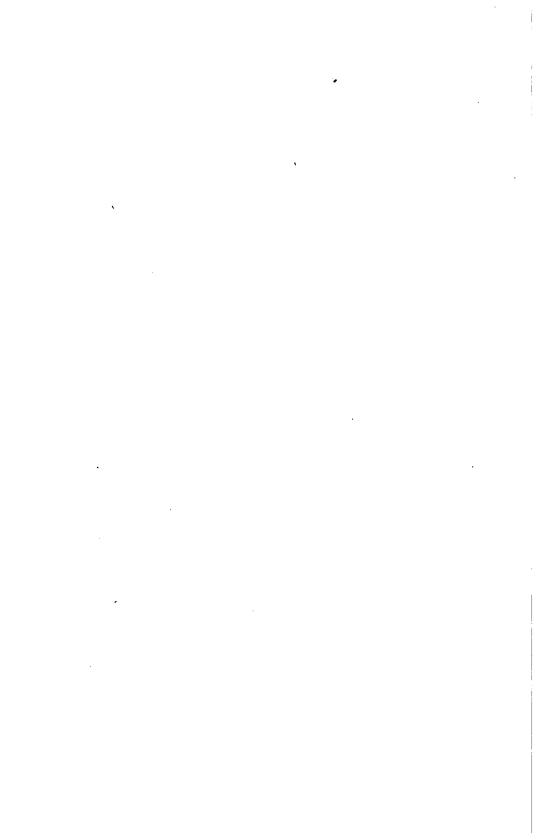
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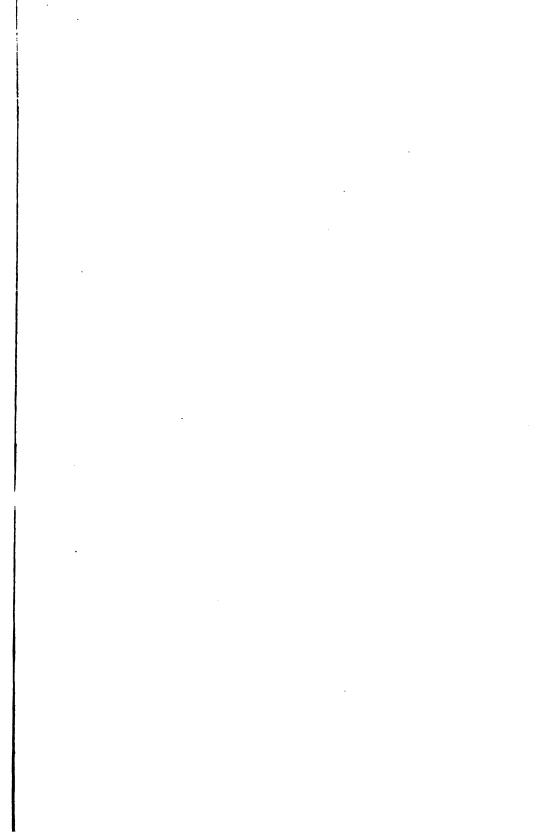
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# Beet-Sugar Making and Its Chemical Control

BY

Y. NIKAIDO, B.Sc., M.A.



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#### PREFACE

The aim of this work is to aid those who are starting their career in the beet-sugar manufacture but lack systematic training in the technique thereof. Having this in view, the author has endeavored to explain not only the practical operations of sugar house stations but also the fundamental principles involved in the various processes of the sugar house and in the methods for analysis of various sugar house products.

Especial attention of the reader is invited to Table IV which may be used to advantage by sugar house chemists in their control work as it dispenses with practically all the calculations pertaining to the determinations of sugar and purity of juices and Brix of undiluted thick syrups.

In preparing this work, the author has made use of the following references:—

An Introduction to the Study of Chemistry, Theoretical Chemistry, Organic Chemistry, Analytical Chemistry, Organic Chemistry, Quantitative Chemical Analysis, Engineering Chemistry, Elementary Treatise on Natural Philosophy, Elementary Treatise on Physics, Botany (Brief Course). Mechanical Engineer's Pocket-Book. Quantitative Estimation of Sugars, Optical Rotation of Organic Substances, Handbook for Beet-Sugar House Chemists, Beet-Sugar Manufacture and Refining. Anleitung zur Untersuchung, Leitfaden fur Zuckerfabriks-Chemiker. Lehrbuch der Zuckerfabrikation, Handbuch der Zuckerfabrikation. Zuckerfabrikation. Chemie der Zuckerarten, Traite de la Fabrication du Sucre,

Remsen. Remsen. Bernthsen. Treadwell. Remsen. Thorpe. Stillman. Deschanel. Ganot. Bessey. Kent. Wein. Landolt-Long. Spencer. Ware. Frühling. Stift. Stammer. Stohmann. Classen. von Lippmann, Beaudet-Pellet Saillard.

The author takes this opportunity to acknowledge the services rendered by Mr. S. P. C. Borson, who made the drawings for the illustrations in this book, and by Mr. R. S. Hiltner who reviewed the manuscript and made valuable suggestions. His gratitude is also due to Mr. Frederick Schlumbohm, who gave much important information on the process of sugar boiling.

Owosso, Michigan, 1908. Y. N.



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# Chapter I.

#### DEFINITIONS OF CHEMICAL TERMS.

Introductory.—It is assumed, in this work, that the readers are familiar with elementary treatises on chemistry. It will not be, however, out of place to review briefly important points in general chemistry in order to introduce the reader to the object of the present work.

The Field of Chemistry.—Chemistry deals with the composition of matter or substances and changes in the composition. Substances may be divided into two classes: elements and compounds.

Elements.—An element is a body which may not be resolved into simpler forms. There are 78 definitely known elements. The following table of elements provided with symbols and atomic weights is taken from the Journal of the American Chemical Society, Vol. XXVII, No 1.

	0 = 16	H = 1		0 = 16	H = 1
Aluminum Al	27.1	26.9	Fluorine F	19.	18.9
AntimonySb	120.2	119.3	Gadolinium Gd	156.	154.8
ArgonA	39.9	39.6	Gallium Ga	<b>7</b> 0.	69.5
Arsenic As	75.0	74.4	Germanium Ge	72.5	72.
BariumBa	137.4	136.4	GlucinumGl	9. 1	9.03
BismuthBi	208.5	206.9	Gold · · · · · · · · Au	197.2	195.7
$Boron \cdot \cdots \cdot B$	11.0	10.9	Helium He	4.	4.
Bromine Br	79.96	79.36	$Hydrogen \cdot \cdot \cdot \cdot H$	1.008	3 I.000
CadmiumCd	112.4	111.6	IndiumIn	115.	114.1
CaesiumCs	132.9	131.9	Iodine · · · · · I	126.97	126.01
CalciumCa	40. I	39.7	IridiumIr	193.0	191.5
Carbon C	12.00	11.91	Iron Fe	55.9	55.5
CeriumCe	140.25	139.2	KryptonKr	81.8	81.2
ChlorineCl	35.45	35.18	LanthanumLa	138.9	137.9
ChromiumCr	52.1	51.7	LeadPb	206.9	205.35
CobaltCo	59.0	58.55	Lithium · · · · · Li	7.03	6.98
Columbium Cb	94.	93.3	Magnesium Mg	24.36	24.18
CopperCu	63.6	63.1	Manganese Mn	55.0	54.6
ErbiumEr	166.	164.8	Mercury Hg	200.0	198.5

#### TABLE OF ELEMENTS—(Continued).

	O = 16	H = I		O = 16	H = I
Molybdenum · · · Mo	<b>9</b> 6.0	95.3	Silver Ag	107.93	107.11
Neodymium · · · · Nd	143.6	142.5	Sodium ·····Na	23.05	22.88
Neon ·····Ne	20.	19.9	Strontium · · · · · Sr	87.6	86.94
Nickel · · · · · Ni	58.7	58.3	SulphurS	32.06	31.82
Nitrogen · · · · · N	14.04	13.93	TantalumTa	183.	181.6
Osmium Os	191.	189.6	Tellurium Te	127.6	126.6
Oxygen O	16.00	15.88	TerbiumTb	160.	158.8
Palladium Pd	106.5	105.7	ThalliumTl	204.I	<b>20</b> 2.6
Phosphorus P	31.0	30.77	ThoriumTh	232.5	230.8
Platinum Pt	194.8	193.4	ThuliumTm	171.	169.7
Potassium · · · · · K	39.15	38.85	Tin · · · · · · Sn	119.0	118.1
Praseodymium .Pr	140.5	139.4	TitaniumTi	48.1	47.7
Radium · · · · Ra	225.	223.3	TungstenW	184.	182.6
RhodiumRh	103.0	103.3	UraniumU	238.5	236.7
RubidiumRb	85.5	84.9	$Vanadium \cdots V$	51.2	50.8
Ruthenium Ru	101.7	100.9	Xenon · · · · · · Xe	128.	127.
Samarium Sm	150.3	149.2	$Ytterbium \cdots Yb$	173.0	171.7
ScandiumSc	44. I	43.8	YttriumYt	89.0	88.3
Selenium ·····Se	79.2	78.6	ZincZn	65.4	64.9
SiliconSi	28.4	28.2	ZirconiumZr	90.6	89.9

Chemical Symbols.—As may be seen from the above table, each element is designated by a symbol, which is usually the initial letter, written in capital, of the Latin name of the element it stands for; for example, O for oxygen and H for hydrogen. In some symbols, a small letter is added to the capital letter so that those elements having the same initial letter may not be confounded; for example, platinum is designated by Pt so that it may be distinguished from phosphorus, which is represented by P.

Compounds.—A compound is a body which is composed of two or more elements united chemically, as, for example, water, which is composed of 2 atoms of hydrogen and 1 atom of oxygen as shown by the formula H<sub>2</sub>O.

Definition of Chemistry.—Chemistry may be defined as the science which investigates systematically the properties of elements, the laws governing the combinations of these elements, and the chemical reactions of compounds.

Atoms.—An atom is the theoretical unit of mass of any element or the smallest particle of elementary bodies which cannot undergo any further subdivision. An atom cannot exist in an isolated condition, but two or more of them combine together forming a body called a *Molecule*.

Atomic Weight.—The atoms of various elements have constant relative weight, the weight of an atom of hydrogen being taken as the unit. The atomic weights under H=1 in the table, as it suggests itself, are calculated on the basis that an atom of hydrogen is taken as 1. Those under O=16 are calculated on the basis that an atom of oxygen is taken as 16. The former are most rational as the element (hydrogen) whose atom has the smallest mass is taken as unity. The latter are of most practical value since they dispose, as may be seen, of many fractional figures, thus simplifying the calculation of chemical problems. Either may be used in computing chemical analytical data. One must, however, adhere to one system, which he has chosen at the start, throughout these calculations.

Molecules.—A molecule is the smallest quantity of an element or compound that exists in an isolated state. It is composed of two or more atoms of the same or different elements. Molecular weight is obtained by adding the weights of all the atoms which constitute a molecule of an element or compound.

Valence.—As may be seen later, hydrochloric acid is represented by the formula HCl, water by H<sub>2</sub>O, sodium chloride (common salt) by NaCl, sulphuric acid by H<sub>2</sub>SO<sub>4</sub>, calcium oxide (lime) by CaO. HCl signifies that hydrochloric acid consists of one atom of hydrogen and one atom of chlorine; H<sub>2</sub>O signifies that water consists of two atoms of hydrogen and one atom of oxygen; NaCl, that sodium chloride consists of one atom of sodium and one atom of chlorine; H<sub>2</sub>SO<sub>4</sub>, that sulphuric acid consists of two atoms of hydrogen, one atom of sulphur, and four atoms of oxygen; CaO, that calcium oxide consists of one atom of calcium and one atom of oxygen. This implies that each of these elements has a certain combining power. Such a power is called the Valence of the Elements. Valence of hydrogen

is taken as unity. An atom of an element that combines with or is replaced by one atom of hydrogen is called *Univalent or a Monad*. Any element whose one atom can combine with or can be replaced by two, three, four, five, or six atoms of hydrogen, is called respectively bivalent, trivalent, tetravalent, pentavalent, or hexavalent and may be regarded as having two, three, four, five, or six bonds by means of which each can combine with other elements. Sometimes they are respectively named diads, triads, tetrads, pentads, or hexads. One atom of sodium may be replaced by one atom of hydrogen, as, for example:

hence sodium is univalent. One atom of oxygen combines with two atoms of hydrogen, like H<sub>2</sub>O (water); hence oxygen is bivalent.

The structure of molecules of any compounds may be explained graphically according to this theory in the following manner: Water, H<sub>2</sub>O:H—O—H;Carbon dioxide,CO<sub>2</sub>:O=C=O;

$$O-H$$
  
 $H-Cl$ ; Sulphuric acid,  $H_2SO_4$ :  $O=S=O$ ; Sodium chloride,  
 $O-H$ 

NaCl: Na — Cl; Calcium oxide, CaO: Ca=O.

The valences of the most commonly known elements are as follows:

Aluminum	Mercury I and 2 Nitrogen
Chlorine I and 5 Copper 2 and I	Phosphorus 3 and 5 Potassium 1
Hydrogen · · · · · · · I	Silver I
Iron 2 and 3	Sodium I
Lead 2	Sulphur 2, 4 and 6.
Magnesium 2	

Inorganic and Organic Chemistry.—Chemistry is divided into two branches: namely, Inorganic and Organic Chemistry. Formerly it was supposed that elements and compounds treated in inorganic chemistry are derived from inorganic bodies, for example, ores and rocks, while the compounds dealt with in organic chemistry are originated from organisms,—plants and animals. It is, however, impossible to draw a distinct line between these branches of chemistry, as the investigations of comparatively recent years show that many compounds included formerly in organic chemistry are not necessarily of organic origin, and that many compounds found in the organs of plants and animals may be prepared in the laboratory. In general, however, it may be said that inorganic chemistry treats of metals, non-metals and their compounds, while organic chemistry deals with compounds of carbon.

## Chapter II.

#### NON-METALLIC ELEMENTS.

Introductory.—Oxygen, nitrogen, carbon, chlorine, bromine, iodine, fluorine, sulphur, phosphorus, boron, and silicon are commonly known as non-metals; of these, oxygen, nitrogen, carbon, sulphur, chlorine, phosphorus, and silicon alone will be discussed in the present work, as the knowledge of the properties of these elements is very important to the sugar-house chemist. Although hydrogen belongs to the group of metals, it should properly be discussed at the outset, as the non-metals named above could not be well considered without a knowledge of hydrogen.

Hydrogen.—Symbol H; Atomic weight I; Valence I. Hydrogen in its normal state is a gas. It combines with various non-metals, forming acids; for example, it forms with chlorine, hydrochloric acid; with sulphur trioxide, sulphuric acid. The simplest method of preparing hydrogen is by dissolving metallic zinc in hydrochloric or sulphuric acid whereupon the zinc replaces the hydrogen of the acid, the gas being evolved vigorously. The equation representing the reaction is:

Hydrogen gas has no taste, color, or odor. It burns in air, combining with oxygen of the atmosphere, forming water.

Oxygen.—Symbol O; Atomic weight 16; Valence 2. Oxygen exists in a free state in the atmosphere, forming about one-fifth of the volume of the same. It also exists combined with other elements forming innumerable compounds. It is present in most of the substances forming the earth's crust. It may be prepared by heating mercury oxide or a mixture of potassium chlorate and manganese dioxide. The reactions may be represented by the following equations:

Mercuric oxide HgO	=	Mercury Hg	+	Oxygen O;
Potassium chlorate KClO <sub>3</sub>	=	Potassium chloride KCl	+	Oxygen 3O.

Oxygen supports combustion. Combustion is simply a chemical combination of oxygen with another element. For example, if we light hydrogen gas, it burns with a blue flame, consuming oxygen in the air, the resulting product being water  $(H_2O)$ . If we heat a piece of watch spring (iron) red-hot and dip it in a bottle containing oxygen, it will burn with a brilliant flame, forming iron oxide  $(Fe_2O_3)$ . A piece of charcoal (carbon) will burn in the atmosphere of oxygen with a bright flame, forming carbon dioxide gas  $(CO_2)$ .

Water.—If we mix two volumes of hydrogen and one volume of oxygen and apply a light to the mixture, it will explode with a loud report, the resulting product being water (H<sub>2</sub>O). Water is a colorless, odorless, tasteless, and transparent liquid, and its reaction is neutral. It is one of the most important and most widely distributed substances in nature, occurring in one or the other of the three physical states,—solid, liquid, or vapor. In oceans, rivers, and lakes, it exists as liquid; in the polar regions, in solid conditions,—ice or snow; in the atmosphere, as vapor. It also occurs in combination in mineral substances and animal and plant bodies.

Water is the most efficient solvent and there are very few substances which are not affected by it. All natural water contains more or less soluble substances dissolved from the strata as it passes through or over them. Rain-water contains a very small amount of ammonia, ammonium nitrate, carbonic acid, nitrous acid, and sulphurous acid, which exist in the atmosphere in minute quantities. Spring- or well-water contains much larger quantities of soluble substances. When the quantity of soluble matter is so large that the water has a peculiar taste or odor, it is called "mineral water." When the amount of soluble substances is so small that it does not affect its taste, it is called "fresh water." Sea-water is a mineral water, as it contains a large amount of soluble matter, its constituents being chlorides and sulphates of sodium, potassium, magnesium, and calcium and minute quantities of silica, bromine, iodine, and phosphoric acid. Ordinary well- or river-water is fresh water, the soluble substances consisting usually of sodium chloride, sodium sulphate, potassium sulphate, calcium carbonate, calcium sulphate, magnesium sulphate, magnesium carbonate, silica, iron oxide, and alumina.

Knowledge of the quality of water as well as of the quantity of soluble substances in water is very important for steam engineering as well as for sugar manufacture. Calcium sulphate, calcium carbonate, and magnesium carbonate are objectionable substances in water for use in boilers, as they incrust tubes and plates, often causing considerable trouble. Magnesium chloride and iron compounds are also injurious to boilers, as they tend to cause corrosion of the boiler plates.

Water containing compounds of calcium is not particularly objectionable for the use in sugar manufacture, for when beet juice, as will be explained later, is treated with lime and the excess of lime is removed by precipitating with carbon dioxide or sulphur gas, compounds of calcium introduced through water may also be separated. Water containing large quantities of compounds of magnesium and iron are very objectionable for the use in sugar manufacture. Compounds of magnesium remain dissolved in sugar solution and prevent crystallization of sugar. Such compounds are called melasgenic, or molasses producing Iron oxide and other iron salts also remain dissolved in sugar solution and give grayish yellow color to sugar crystals. For the above reasons, it is advisable to investigate the quality of water before the location of a sugar factory is decided upon. The following are the results of an analysis of water taken from the well at Ames, Nebraska, at a depth of 27 feet, on July 21, 1905.

	Parts per 100,000	Grains per U.S. on
Sodium chloride	.8617	· <b>499</b> 4
Potassium sulphate	2.0641	1.2034
Sodium sulphate	1.9490	1.1363
Sodium carbonate	3.7515	2.1871
Calcium carbonate	15.8803	9.2582
Magnesium carbonate		2.0299
Iron oxide and alumina	.8000	.4664
Silica		1.7432
Organic and volatile matter	.0889	.0518
Total soluble matter	31.8673	18.5757

This water is regarded fairly good for use in sugar manufacture.

Nitrogen.—Symbol N; Atomic weight 14; Valence 3 and 5. Nitrogen is a colorless, tasteless, and odorless gas. It does not support combustion and sustains no life. It constitutes fourfifths of the atmosphere. Usually this gas is prepared by burning phosphorus under an inverted bell-jar placed over water, whereupon all the oxygen in the enclosed air is consumed while nitrogen remains intact. When the white fumes of phosphorus pentoxide are all dissolved in the water, the gas remaining in the jar is nearly pure nitrogen. Nitrogen enters into a great variety of combinations. The most important nitrogen compounds are ammonia, oxides of nitrogen, nitrates and nitrites of various bases, proteids, and alkaloids. Only nitric acid and ammonia need to be considered here, as the sugar chemist has occasion to deal with the former in analytical work and with the latter during the purification of the beet juice.

Nitric Acid.—Formula HNO<sub>3</sub>; Molecular weight 63. Nitric acid is commonly prepared by distilling a mixture of sulphuric acid and sodium nitrate, the equation being:

Nitric acid is a very corrosive, poisonous, pungent liquid, which boils at 86°C and solidifies at —47°C. It dissolves most metals, forming nitrates and nitric oxide (NO); for example, nitric acid dissolves silver, forming silver nitrate and nitric oxide:

It reacts with oxides, hydroxides, and carbonates of all metals, forming nitrates. Nitric acid and nitrates are extensively used in analytical work, art, and medicine. Sodium nitrate (Chili Saltpeter) is extensively used as a fertilizer, since nitrogen is one of the most important plant-foods.

Ammonia.—Formula H<sub>3</sub>N; Molecular weight 17. As the formula H<sub>3</sub>N indicates, ammonia consists of one atom of nitrogen

and three atoms of hydrogen. It is a colorless gas under ordinary pressure and temperature, with a pungent odor and a strongly alkaline reaction, and is very soluble in water.

Alkalinity may be tested with litmus paper or with a drop of a solution of phenolphthalein in alcohol. Any alkaline substance turns red litmus blue or changes the color of phenolphthalein solution from colorless to pink.

Ammonia dissolves in, or rather, combines with water, forming ammonium hydroxide (H<sub>4</sub>NOH), the reaction being:

Ammonia Water Ammonium hydroxide 
$$H_2N + H_2O = H_4NOH$$
.

In the chemical laboratory, ammonia may be prepared by heating a mixture of one part by weight of ammonium chloride (H<sub>4</sub>NCl) and two parts by weight of quicklime (CaO), the reaction being:

Ammonium chloride Lime Ammonia Calcium chloride Water 
$$2H_4NCl + CaO = 2H_3N + CaCl_2 + H_2O$$
.

Sources of Ammonia.—When organic substances which contain nitrogen and hydrogen, such as leather, wool, or gelatine, are decomposed, some of the nitrogen and hydrogen unite to form ammonia. If we heat a piece of wool or silk in a small test tube, it becomes charred and gives off water and gas. This gas has been shown to be partly ammonia and it will turn red litmus paper blue. When we boil the materials mentioned above with strongly alkaline substances, such as lime or caustic soda, ammonia is formed.

Ammonia in Beet Juice.—There is ammonia everywhere in the steam lines and condensed water of a sugar factory. This may be traced back to the composition of sugar beets. As will be described under Constituents of Non-Sugars, beet juice contains a large number of nitrogenous compounds. Some of these compounds are precipitated when the beet juice is treated with lime, but albumen and a few others remain in the juice; when boiled in the evaporators and vacuum pans, these compounds are partly converted into ammonia which vaporizes

readily with steam and partially condenses with water. Condensed water from the evaporators is used for boiler feed, consequently ammonia is found in all steam lines and condensed waters of a sugar factory. The amount, however, is not sufficient to do any perceptible injury except to brass and copper pipe fittings, and so forth.

Difficulties Caused by Ammonia in the Juice.—Ammonia in the juice sometimes causes trouble. As will be explained later, the beet juice is treated with lime and the excess thereof is precipitated twice with carbon dioxide gas and finally with sulphur dioxide gas. Throughout the process of purification of the juice, a certain amount of lime is kept in it, the amount varying at different stages. When it leaves the sulphur station and enters the evaporators, it contains about 0.01 per cent of lime (CaO). The amount of lime is tested with a standard acid solution. (The process will be described later). This test, however, simply indicates total alkalies, which may consist of lime (CaO), ammonia, (H4NOH) and hydroxides of sodium and potassium (NaOH and KOH). Sometimes, the juice contains a considerable quantity of ammonia, but as we cannot estimate ammonia in a short time, we assume that the entire alkaline reaction of the juice is due to lime. This assumption, however, often leads us to errors, for such juice, sometimes, loses alkalinity entirely after evaporation, undoubtedly due to the evaporation of ammonia. This transformation of the juice sometimes does not stop at the neutral point, but it becomes distinctly acid. why the juice does not remain neutral instead of becoming acid after evaporation of ammonia is an open question. The author, however, is of the opinion, that although the acidity of the juice may be partially caused by the formation of new acids as decomposition products of invert sugar and other organic compounds, it is largely due to the hydrolysis of some of the ammonia salts of organic acids, as, for example, ammonium succin-

ate, which has the formula, C,H, COOH,N, when boiled in water

for sometime, may liberate one molecule of ammonia, the resulting

In fact, if we boil a solution of neutral ammonium succinate for some time, it becomes distinctly acid.

Whether the acidity of the juice caused by the liberation of ammonia is injurious to sugar or not, the author is not able to make a definite statement, although he observed a slight increase of invert sugar after such juice has been concentrated by boiling down in a porcelain evaporator on a hot water bath. The presence of proper quantity of lime (CaO) always helps to improve the quality of the juice, therefore it is advisable to increase the alkalinity of the juice before evaporation in case the juice tends to become acid after evaporation. This may be controlled at the second carbonatation and sulphur stations.

Carbon.—Symbol C; Atomic weight 12; Valence 2 and 4. Carbons occurs in nature in three allotropic states:—diamond, graphite, and coal. The first two are crystalline, and the third, amorphous. Diamond crystallizes in octahedron or related forms. It is transparent, colorless, yellow, pink, or blue. It is the hardest substance known, infusible, and a non-conductor of electricity. It burns in oxygen gas at white heat, forming carbon dioxide gas. Graphite is gray-black, crystallizes in six-sided prisms and is a good conductor of electricity. Amorphous carbon occurs in a more or less pure state in lamp-black, wood-charcoal, coal, coke, and animal bone-black. It burns in air at red heat forming carbon dioxide gas (CO<sub>2</sub>). Oxygen combines with carbon forming two oxides of carbon:—carbon monoxide and carbon dioxide.

Carbon Monoxide.—Formula CO; Molecular weight 28. Carbon monoxide is producd by passing carbon dioxide over red hot charcoal or by heating oxalic acid with sulphuric acid. It is colorless, inodorous, tasteless, insoluble in water and is ex-

tremely poisonous. It burns with a light blue flame, forming carbon dioxide.

Carbon Dioxide.—Formula CO<sub>2</sub>; Molecular Weight 44. Carbon dioxide, or carbonic acid is a colorless gas 1.5 times as heavy as air. It is a product of combustion or fermentation. It constitutes on an average 0.04 per cent, by volume, of the air. It has an acid reaction on litmus paper. It does not support combustion nor sustain animal life. Plants absorb the gas, retaining the carbon and liberating oxygen in the sun light. This gas is contained in spring water and helps to dissolve carbonates of calcium and magnesium and iron oxide.

Carbon dioxide is prepared by adding any acid to any carbonate. For example, if we add hydrochloric acid to a solution of sodium carbonate (Na<sub>2</sub>CO<sub>8</sub>), carbon dioxide gas is liberated, leaving sodium chloride in solution. The reaction may be represented by the following equation:

This gas may also be prepared by strongly heating carbonates of alkaline earth metals (calcium, strontium, barium) or heavy metals. If we heat calcium carbonate strongly, it is decomposed into two compounds:—calcium oxide and carbon dioxide; the reaction being:

Calcium carbonate		Calcium oxide		
CaCO <sub>3</sub>	=	CaO	+	CO2.

The process of making lime (CaO) and carbon dioxide is based on this principle and will be discussed under CALCIUM.

Carbon dioxide gas very readily combines with oxides and hydroxides of alkaline metals (sodium, potassium, and lithium), forming soluble carbonates and with those of alkaline earth metals (calcium, strontium, and barium) forming carbonates insoluble in water. As will be discussed later, the beet juice is treated with a large quantity of lime in order to decompose or precipitate the impurities therein, and it is customary to remove the excess of lime content by precipitating with carbon dioxide gas, as the

latter combines with the former according to the following equation:

This process of precipitation is called saturation or carbonatation. Carbonatation is the reverse of the process of making lime and carbon dioxide; for, in the latter, calcium carbonate is decomposed into lime and carbon dioxide by strongly heating, while, in the former, lime and carbon dioxide are brought together so that calcium carbonate may be formed again. It is claimed that carbon dioxide, besides being a precipitant of lime in the juice, improves the quality of the juice by precipitating some impurities such as calcium oxalate and other organic salts of calcium.

Chlorine.—Symbol Cl; Atomic weight 35.5; Valence I. Chlorine is a yellowish green gas, has a powerful irritating smell, and attacks the mucous membranes of the throat and lungs. It is prepared by heating a mixture of hydrochloric acid and manganese dioxide. The reaction may be represented by the following equation:

It combines with hydrogen with explosive violence in direct sunlight or when a light is applied. It is very soluble in water. The solution forms hydrochloric acid in sunlight, liberating oxygen. It has a powerful bleaching property. It occurs in combination with various bases, such as sodium chloride (common salt), potassium chloride, or calcium chloride. If we add silver nitrate to a solution of any chloride, silver chloride is formed, which is insoluble in any acid. It is on this account that chlorine occurring in combination with bases is determined as silver chloride. The method for determination will be described in the chapter on Special Analysis.

Hydrochloric Acid.—Formula HCl; Molecular weight 36.5. Hydrochloric acid is a colorless, pungent, irritating gas. It is incombustible, extinguishes flame, and liquefies under 40 atmos-

pheric pressure at 10°C. Water dissolves 450 volumes of hydrochloric acid at an ordinary temperature. Hydrochloric acid is prepared by heating a mixture of sodium chloride (common salt) and sulphuric acid, the reaction being:

Acid.—Hydrochloric acid is a typical representative of a class of compounds known as acids, hence it would be very convenient to discuss here general characteristics of acids. Alkali and salt will also be discussed here as they will be frequently referred to in discussion of acids. An acid reddens blue litmus paper or solution. It generally dissolves metals; for example, hydrochloric acid dissolves zinc, forming zinc chloride (ZnCl<sub>2</sub>) and hydrogen, the reaction being:

It also dissolves carbonates, hydroxides, and oxides of alkaline-metals (sodium, potassium, and lithium), alkaline earth metals (calcium, strontium, and barium), and heavy metals (zinc, iron, tin, and so forth). These reactions may be illustrated by the following equations:

Alkali.—Oxides and hydroxides of alkaline metals, and alkaline earth metals are called alkalies. They turn red litmus paper or solution blue, phenolphthalein solution pink, and turmeric paper brown. They all have power to neutralize acids.

Salt.—When a certain amount of acid is added to a certain amount of any alkaline compound, both lose their characteristic reactions, becoming indifferent to litmus paper or phenolphthalein solution. This reaction is called neutral and the product, neutral salt. For example, if we add a certain amount of hydrochloric acid to a certain amount of sodium hydroxide, sodium chloride, or common salt, which is perfectly neutral, is formed.

Chemical Equivalents.—The amount of each substance to be taken in order to produce a neutral salt is governed by chemical equation. An examination of the equation

Hydrochloric acid		Sodium hydroxide		Sodium chloride		Water
HCl	+	NaOH	=	NaC1	+	H <sub>2</sub> O

indicates that one molecule of hydrochloric acid neutralizes one molecule of sodium hydroxide, producing one molecule of the neutral salt, sodium chloride and one molecule of water. The molecular weight of hydrochloric acid being 36.5, that of sodium hydroxide 40, that of sodium chloride 58.5, and that of water 18, we can deduce from the above equation, that 36.5 parts by weight of hydrochloric acid neutralize 40 parts by weight of sodium hydroxide, producing 58.5 parts by weight of sodium chloride and 18 parts by weight of water. These quantities are called chemical equivalents.

The fact that a definite quantity of an acid neutralizes a definite quantity of an alkali or vice versa suggests that if the strength of one be known, that of the other can be found and that such a principle may be applied for analytical purposes.

Alkalimetry.—If we have a solution which contains exactly 36.5 grams of hydrochloric acid in one liter and a solution of sodium hydroxide whose strength is not known, we can find the strength of the latter by neutralizing it with the former. It should be noted that one cubic centimeter (c.c.) of this acid solution contains 0.0365 gram of hydrochloric acid. Fill a burette of 50 c.c. capacity exactly to the zero mark with the acid solution. Measure exactly 20 c.c. of the sodium hydroxide solution of unknown strength, transfer it to a small porcelain evaporating dish and add a few drops of phenolphthalein solution, which

will instantly give a pink color to the solution. Now add very carefully the acid solution from the burette, stirring continually until the pink color just disappears. At this point, all the alkali has been neutralized by the hydrochloric acid. Note the number of cubic centimeters of the acid required to reach this point. Assume that 25 c.c. of the acid were required, then, as I c.c. contains 0.0365 gram of hydrochloric acid, 20 c.c. of the alkali solution required 25×0.0365=0.9125 gram of the hydrochloric acid. As stated previously, the chemical equivalent of hydrochloric acid to sodium hydroxide is 36.5 to 40.0, therefore, the amount of sodium hydroxide equivalent to 0.9125 gram of hydro-

chloric acid would be 0.9125  $\times \frac{40.0}{36.5} = 1.00$  gram. Hence, we can

deduce that 20 c.c. of the alkali solution contained one gram of sodium hydroxide. This method of estimating alkaline substances is known as alkalimetry and is applied for the estimation of any alkalies.

Acidimetry.—The method described above may be reversed, that is, by the use of sodium hydroxide solution of known strength, an acid solution of unknown strength may be determined. Fill a 50 c.c. burette with a solution whose I c.c. contains exactly 0.040 gram of sodium hydroxide to the zero mark; measure into a porcelain evaporating dish, 25 c.c. of dilute hydrochloric acid solution of unknown strength, and add a few drops of phenolphthalein solution and then, the sodium hydroxide solution from the burette, a little at a time, stirring continually until a faint pink color appears. Note the number of cubic centimeters of the sodium hydroxide solution required to reach this point. Assume that 26.5 c.c. of the sodium hydroxide were required, then the total amount of sodium hydroxide required would be  $26.5 \times 0.040 = 1.06$  grams. The chemical equivalent of sodium hydroxide to hydrochloric acid being 40 to 36.5, 1.06 grams

of sodium hydroxide would be equivalent to 1.06  $\times \frac{36.5}{40} = 0.9672$ 

gram of hydrochloric acid. Hence, 25 c.c. of the dilute acid solution contained 0.9672 gram of pure hydrochloric acid. This

method of estimating the strength of acid solutions is called ACIDIMETRY.

Definition of Normal Solution.—It will be of advantage to explain the terms Monobasic and Dibasic prior to discussing normal solutions. A compound containing one atom of hydrogen or its equivalent of another base-forming element as base is called Monobasic. Hydrochloric acid is monobasic as it contains one atom of hydrogen as base. Sodium hydroxide is also monobasic as it contains one atom of sodium, which is equivalent to one atom of hydrogen, as base. A compound containing two atoms of hydrogen or their equivalent of another base-forming element as base, is called Dibasic. Sulphuric acid is dibasic as it contains two atoms of hydrogen as base. Sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) is also dibasic as it contains two atoms of sodium, which are equivalent to two atoms of hydrogen, as base.

A solution of known strength is called a standard solution. It is not absolutely necessary to have a standard solution of any particular strength. It is, however, customary to prepare, for a standard solution, such a solution that one liter thereof contains one molecular weight in grams of any monobasic compound. Such a solution is called *Normal Solution*. For a normal solution of dibasic compounds, one-half the molecular weight in grams is taken for one liter. It is evident, then, that any normal solution is so made that one liter thereof contains one gram of hydrogen or its equivalent of any other base-forming element combined as base.

Normal Hydrochloric Acid Solution.—As stated previously, the molecular weight of hydrochloric acid is 36.5, hence, normal hydrochloric acid solution is such a solution that one liter thereof contains 36.5 grams of pure hydrochloric acid.

Normal Sodium Hydroxide Solution.—As the molecular weight of sodium hydroxide is 40, one liter of normal sodium hydroxide solution contains 40 grams of pure sodium hydroxide.

Normal Sulphuric Acid Solution.—As the molecular weight of sulphuric acid is 98 and it is dibasic acid, normal sulphuric

acid solution is so made that one liter thereof contains one-half this quantity, namely,  $\frac{98}{2} = 49$  grams of pure sulphuric acid.

Methods for Preparation of Normal Solutions.—There are several methods for the preparation of normal solutions, but those that require long manipulation will not be discussed here as great accuracy is not required in sugar house control work. One of the simplest and fairly accurate methods is the use of pure oxalic acid (C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>) or potassium tetraoxalate (C<sub>4</sub>H<sub>3</sub>KO<sub>8</sub>+2H<sub>2</sub>O) for standardization of normal sodium hydroxide solution which could in turn be used for standardizing any other normal acid solutions. This method will be described in this work.

Normal Oxalic Acid Solution.—As the chemical formula

sists of two atoms of carbon, four atoms of oxygen, and two atoms of hydrogen. As these two atoms of hydrogen are combined as base, oxalic acid is regarded as a dibasic acid. Its molecular weight is 90. In order to prepare normal solution

of oxalic acid, therefore, half the molecular weight, 
$$\frac{90}{2}$$
=45 grams

should be taken for one liter. This, however, applies only when oxalic acid is freed from water of crystallization by drying at  $100^{\circ}$ C to constant weight. When crystallized from water, one molecule of oxalic acid takes up two molecules of water as water of crystallization, as may be seen from the formula  $C_2H_2O_4+2H_2O$ ; hence, when crystallized oxalic acid is to be

used,  $\frac{90+36}{2}$  = 63 grams should be taken for one liter of normal solution.

Normal Potassium Tetraoxalate Solution.—In standardizing an alkali solution, potassium tetraoxalate solution is preferred to oxalic acid as the former may be obtained in purer state than the latter. Potassium tetraoxalate is an acid potassium COOH COOH salt of oxalic acid, having the chemical formula | + | + COOH COOK

2H<sub>2</sub>O, and its molecular weight, including water of crystallization, is 254.206. As the formula indicates, there are three atoms of hydrogen combined as base in one molecule of this compound; hence, one-third the molecular weight, namely,

 $\frac{254.206}{3}$  = 84.7353 grams should be taken for one liter of normal solution of this compound.

. Since normal solution is too strong for accurate analytical work, a solution with one-tenth normal strength is used in ordinary analytical work; such a solution is called *Decinormal* 

or Tenth-Normal Solution and is designated by  $\frac{N}{10}$ . 1000 c.c. tenth-normal solution of potassium tetraoxalate contains

8.47353 grams of pure crystallized potassium tetraoxalate. 1000 c.c. of tenth-normal oxalic acid solution contains 6.3024 grams of pure crystallized oxalic acid.

Precautions Required in Manipulation in Preparation of Normal Solutions.—In the manipulation of chemical analysis, the apparatus should be kept absolutely clean; beakers, burettes, pipettes, and flasks should be washed first with hydrochloric acid, then with ordinary water and finally rinsed twice with distilled water before they are used. The temperature of the room should be kept as constant as possible. All standard solutions should be made at the average temperature of the room.

Preparation of Notation of Potassium Tetraoxalate.—

Potassium tetraoxalate obtained from chemical dealers as chemically pure is usually reliable. It is, however, likely that some of the crystals may have lost their water of crystallization and it is, therefore, safest to recrystallize it just before making a standard solution therefrom. Dissolve completely about 20

grams of potassium tetraoxalate in 50 c.c. of distilled water in a perfectly clean beaker, filter the solution into another clean beaker. After filtering, evaporate the solution on a water bath until a drop thereof on a watch glass shows fine crystals on cooling. Then set the beaker aside and allow the solution to cool and yield crystals. When a good crop of the crystals is obtained, pour out the supernatant solution and dry the crystals between filter papers until all the moisture has been absorbed, then transfer into an air tight bottle.

Weigh out exactly 8.4735 grams of the potassium tetraoxalate just recrystallized on a watch glass as quickly as possible, since it may lose some of the water of crystallization on coming in contact with dry air. Dissolve it in a small quantity of distilled water in a perfectly clean beakthe solution is completed, er. transfer a clean one-liter flask and rinse the beaker several times into the flask. Now add more water until the bottom of the meniscus (when the surface of a liquid contained in a tube assumes a form of concave hemispherical segment, it is called meniscus) agrees with the 1000 c.c. mark and then mix thoroughly by shak-

ing. This is 
$$\frac{N}{10}$$
 solution of potassium tetraoxalate.

Preparation of Normal Sodium Hydroxide Solution.—In preparing normal sodium hydroxide solution, chemically pure stick caustic soda should be used, care being taken to select clear looking sticks and to reject all the weathered portions. Weigh approximately 41 grams of the selected sticks, dissolve in a small quantity of distilled water in a clean beaker. When the solution is completed, transfer it to a clean graduated one-liter flask, and rinse the beaker into the flask several times. Then add more distilled water to complete the volume of the solution to the one-liter mark and mix thoroughly by shaking. In order to determine the exact strength of this solution, transfer, by means of a pipette, exactly 10 c.c. of this solution to a clean 100 c.c. flask, rinse the pipette into the flask, complete the volume to the 100 c.c. mark with distilled water and mix thoroughly.

Now fill a 50 c.c. burette with this solution exactly to the zero mark, another with the  $\frac{N}{10}$  solution of potassium tetraoxalate al-

ready made. Draw about 20 c.c. of the potassium tetraoxalate solution into a small porcelain evaporating dish, add a few drops of phenolphthalein solution, which is made by dissolving one gram of phenolphthalein in 100 c.c. of 50 per cent alcohol, and then cautiously add the sodium hydroxide solution from the burette, stirring constantly until a faint pink color appears. In case an excess of sodium hydroxide is added, some more potassium tetraoxalate solution should be added, ceasing at the point where a faint pink color remains permanent. Note the number of c.c. of each solution required. Repeat this operation, which will be called *titration* hereafter, several times and take the average for final calculation. This may be best explained by an example:

Sodiur	n Hydroxide c.c.		Potassium Tetraoxalate c.c.
	19.85	required	20.35
	19.63	- "	20,20
	19.53	"	20.00
Average	19.67	**	20.183
Relative Strength	0.9746	"	1.0000

From the above, it is evident that 974.6 c.c. of the sodium hydroxide solution require 1000 c.c. of the potassium tetra-oxalate solution. Hence, if we take 974.6 c.c. of the sodium hydroxide solution and make it up to 1000 c.c. with distilled water or add 25.4 c.c. of water to 974.6 c.c. of the sodium hydroxide solution, I c.c. of this new solution will neutralize

exactly 1 c.c. of the  $\frac{N}{10}$  solution of potassium tetraoxalate. The

amount of water to be added has been calculated for the sodium hydroxide solution diluted ten times. This calculation can also be applied for the undiluted solution or the solution prepared by dissolving 41 grams of sodium hydroxide in 9000 c.c. of water, that is, the strength of the solution prepared by diluting 974.6 c.c. of the undiluted solution with 25.4 c.c. of water would

exactly correspond to normal solution of potassium tetraoxalate. Measure out, therefore, 974.6 c.c. of the undiluted solution and make it up to 1000 c.c. with water or add 25.4 c.c. of water to 974.6 c.c. of the undiluted solution and mix thoroughly. This new solution is called *Normal Sodium Hydroxide Solution*. Should the quantity of the undiluted solution on hand happen to be greater or less than 974.6 c.c., the amount of water to be added may be calculated by a proportion. Assume the amount of the undiluted solution on hand to be 990 c.c., then the amount of water to be added would be

$$25.4 \times \frac{990}{974.6} = 25.8 \text{ c.c.}$$

In order to test the accuracy of this new solution, transfer 10 c.c. thereof to a 100 c.c. flask and make it up to the 100 c.c.

mark with water and titrate it against  $\frac{N}{10}$  solution of potassium

tetraoxalate. They should correspond exactly if the manipulations have been carried out properly. In case it is found to be too strong, add a calculated amount of water. Should it happen that the solution is weaker, add a small piece of caustic soda

and repeat the entire process. Prepare about 500 c.c. of  $\frac{N}{10}$  solution of sodium hydroxide for future use.

Normal Hydrochloric Acid Solution.—Having completed the preparation of normal sodium hydroxide solution, any normal acid solution may be prepared from this solution. In order to make exact normal hydrochloric acid solution, an approximately normal hydrochloric acid solution should first be made. For finding the number of c.c. of a concentrated hydrochloric acid to be taken for 1000 c.c. of normal hydrochloric acid solution, the

following formula may be used:  $\frac{N.W.}{D \times P.C.}$  = number of c.c. of con-

centrated hydrochloric acid, in which N. W. represents the weight of pure hydrochloric acid required for one liter of normal solution, D, the specific gravity of the concentrated hydrochloric

solution drawn.

final calculation.

acid solution to be used, P. C., the percentage of pure hydrochloric acid contained in the acid solution at that specific gravity. As the molecular weight of hydrochloric acid is 36.5, the weight of pure hydrochloric acid to be taken for one liter of normal solution would be 36.5 grams. The usual specific gravity of the concentrated hydrochloric acid used in chemical laboratories is 1.20 and it contains 40.777 per cent of pure hydrochloric acid; hence, the number of c.c. of this acid to be taken for one liter

of normal solution would be 
$$\frac{36.5}{1.20\times0.40777}$$
 = 74.6 c.c.

It is convenient, however, to make it a little stronger as water may be added after an accurate determination by titrating against  $\frac{N}{10}$  sodium hydroxide solution, therefore measure about 76 c.c. of the acid into a graduated one liter flask, make it up to 1000 c.c., and mix thoroughly by shaking. Transfer 10 c.c. of the solution just prepared to a 100 c.c. flask and make it up to 100 c.c. Fill a 50 c.c. burette with this solution exactly to the zero mark and another with the  $\frac{N}{10}$  sodium hydroxide solution. Draw about 20 c.c. of the acid solution into a small porcelain evaporating dish, add a few drops of phenolphthalein solution, and then cautiously add the  $\frac{N}{10}$  solution of sodium hydroxide, stirring continually until a faint pink color appears. In case an excess of the alkali is added, it should be titrated back with the acid solution. Note the number of c.c. of each

Repeat the titration and take the average for

		EXAMPLE.	
\$	Sodium hydroxide c.c.	Ну	drochloric acid
	20.60	Required	19.80
	21.40	"	20,60
	20.00	" .	19.27
Average	20.67		19.89

If we divide the number of c.c. of the hydrochloric acid solution by the number of c.c. of the sodium hydroxide solution, we obtain the number of c.c. of the hydrochloric acid solution required for neutralizing 1 c.c. of the  $\frac{N}{10}$  sodium hydroxide solution; that is,  $\frac{19.89}{20.67}$ =0.9623 c.c. of the hydrochloric acid solu-

tion is required for neutralizing 1 c.c. of the  $\frac{N}{10}$  solution of so-

dium hydroxide. It is evident, then, that 962.3 c.c. of the acid would correspond to 1000 c.c. of the alkali, or if we dilute 962.3 c.c. of the acid solution to 1000 c.c. with water, the strength of

this solution would exactly correspond to that of the  $\frac{N}{10}$  solution

of sodium hydroxide. If the strength of each of the solutions were ten times as great, their relative strength would be just the same; measure, therefore, exactly 962.3 c.c. of the hydrochloric acid solution, which was made by diluting 76 c.c. of the concentrated hydrochloric acid to 1000 c.c. into a graduated one-liter flask and make it up to 1000 c.c. with water or add exactly 37.7 c.c. of water to 962.3 c.c. of the acid and mix thoroughly. This new solution is called *Normal Hydrochloric Acid Solution*. In case the number of c.c. of the acid solution to be diluted is greater or less than 962.3 c.c., the amount of water to be added may be calculated by a proportion. Assuming that the number of c.c. of the acid on hand to be diluted is 990 c.c., the amount of water

to be added would be  $37.7 \times \frac{990}{962.3} = 38.8$  c.c. In order to determine the accuracy of the new solution, transfer exactly 10 c.c. to a 100 c.c. flask, make it up to 100 c.c. with water and titrate it against the  $\frac{N}{10}$  solution of sodium hydroxide.

Standard Acid Solution Used for Examination of Alkalinity of the Juice.—As will be discussed under calcium and beet juice,

beet juice is treated with a large quantity of lime and an excess thereof is precipitated with carbon dioxide gas. A small quantity of lime, however, is kept in the juice throughout the process. This is called alkalinity of the juice. The amount of lime or degree of alkalinity of the juice varies at different stages of the process and is controlled by chemical tests. Any acid solution of weak strength would answer to this purpose. It is, however, convenient for this purpose to use an acid solution of such a strength that I c.c. thereof will neutralize 0.001 gram of lime (CaO), hence, the principle and method for preparing such an acid solution will be discussed here somewhat in detail. As the chemical equation

indicates, two molecules of hydrochloric acid neutralize one molecule of calcium oxide (lime), hence,  $2\times36.5=73$  parts of hydrochloric acid will neutralize 56 parts of lime or 36.5 grams of hydrochloric acid will neutralize 28 grams of lime. As the strength of the acid solution we seek is such that I c.c. neutralizes 0.001 gram of lime or that 1000 c.c. correspond to I gram of lime, normal hydrochloric acid solution, 1000 c.c. of which neutralize 28 grams of lime, is 28 times as strong as the strength of the solution we seek. Dilute, therefore, one liter of normal hydrochloric acid solution with 27 liters of distilled water in order to obtain a solution I c.c. of which will neutralize 0.001 gram of lime. This solution may be called Twenty-eighth Nor-

$$mal\left(\frac{N}{28}\right)$$
 Hydrochloric Acid Solution. In case a smaller quan-

tity of such a solution is needed, calculate the number of c.c. of normal hydrochloric acid solution required to make 1000 c.c. of such a solution from the following consideration: I c.c. of normal hydrochloric acid solution corresponds to 0.028 gram of lime, and 1000 c.c. of the solution, I c.c. of which neutralizes 0.001 gram of lime, corresponds to I gram of lime, hence, the

number of c.c. of normal hydrochloric acid solution to be taken for making 1000 c.c. of the solution, I c.c. of which neutralizes 0.001 gram of lime, would be  $\frac{I}{0.028} = 35.7$  c.c. Transfer, therefore, exactly 35.7 c.c. of normal hydrochloric acid solution to a graduated 1000 c.c. flask and complete the volume with distilled water.

Standard Alkali Solution Used for Examination of Acidity of the Juice.—The juice, sometimes, becomes acid by over-sulphuring or through the evaporation of ammonia or the formation of new organic acids. In order to examine the acidity, it is necessary to prepare a solution of sodium hydroxide whose strength corresponds with that of the acid solution just made. I c.c. of such a solution represents 0.001 gram of lime. This is made by diluting 35.7 c.c. of the normal sodium hydroxide solution to 1000 c.c. with distilled water.

Normal Sulphuric Acid Solution.—The general characteristics of sulphuric acid will be discussed in the article on SULPHUR, but since the method of preparation of normal sulphuric acid solution is very similar to that of normal hydrochloric acid solution, and as it would be easier for the readers to follow similar lines of thought one after another, it will be discussed here.

The molecular weight of sulphuric acid is 98; it is dibasic acid, that is, one molecule of sulphuric acid contains two atoms of hydrogen combined as base. Hence, according to the definition of normal solution, the weight of pure sulphuric acid to be taken for making 1000 c.c. of normal sulphuric acid solution

would be one-half the molecular weight, namely,  $\frac{98}{2}$  = 49 grams. Approximately 49 grams of pure sulphuric acid may be obtained by taking a number of c.c. of concentrated sulphuric acid calculated from the formula  $\frac{N.W.}{D \times P.C.}$ . Assuming that the specific gravity of the concentrated sulphuric acid to be used is 1.84,

tion.

and the percentage of pure sulphuric acid contained in the same is 97, the number of c.c. of the sulphuric acid to be taken would be  $\frac{49}{1.84 \times 0.97}$  = 27.5 c.c. It is convenient to make it a little stronger so that it may be diluted to the strength we desire after an accurate determination. Measure about 20 of sulphuric acid of the specific gravity 1.84 and place in a graduated 1000 c.c. flask (have the flask about half full of water before adding the acid to prevent overheating and spattering), make it up to 1000 c.c. with distilled water after cooling and mix by shaking. Transfer exactly 10 c.c. of this solution to a 100 c.c. flask by means of a pipette, rinse the pipette thoroughly into the flask and complete the volume to 100 c.c. with distilled water and mix thoroughly. Fill a 50 c.c. burette to the zero mark with this solution and another with the  $\frac{N}{r_0}$  solution of sodium hydroxide. Draw about 20 c.c. of the sulphuric acid solution into a small porcelain evaporating dish, add a few drops of phenolphthalein solution and then, carefully, the  $\frac{N}{N}$  solution

of sodium hydroxide until a faint pink color remains permanent. Note the number of c.c. of each solution required. Repeat the process several times and take the average for the final calcula-

	EXAM	PLE.
Sı	alphuric Acid c.c.	Sodium Hydroxide c.c.
	20.00	20.70
	19.60	20.30
	21.00	21.80
•		
Average	20.20	20.93
Relative Strength	0.9656	1.00

The relative strength indicates that 0.9656 c.c. of the sulphuric acid neutralizes exactly 1 c.c. of the  $\frac{N}{10}$  sodium hydroxide solution. It is evident, then, that a solution made by diluting 965.6 c.c.

of this solution with 34.4 c.c. of distilled water would agree with the  $\frac{N}{10}$  solution of sodium hydroxide, c.c. to c.c. tion of sulphuric acid, whose strength is ten times as great, would bear the same relation to the normal solution of sodium hydroxide, therefore, a solution prepared by diluting 965.6 c.c. of the undiluted acid solution, that is, the solution which was made by diluting 29 c.c. of concentrated sulphuric acid to 1000 c.c., with 34.4 c.c. of distilled water, should agree c.c. to c.c. with the normal solution of sodium hydroxide. This new solution is Normal Sulphuric Acid Solution. In case, the number of c.c. of the undiluted acid solution is greater or less than 965.6 c.c., the number of c.c. of water to be added may be calculated by Assuming the number of c.c. of the undiluted solution on hand to be 900 c.c., the number of c.c. of water to be added would be 34.4  $\times \frac{990}{965.6} = 35.3$  c.c. The accuracy of the solution just prepared may be tested by diluting 10 c.c. of this solution to 100 c.c. with water and titrating it against the  $\frac{N}{10}$  solu-

Standard Sulphuric Acid Solution Used for Examination of Alkalinity of the Juice.—For testing alkalinity of a juice, a standard solution of sulphuric acid is preferred to that of hydrochloric acid, owing, probably, to the fact that the former is a little more stable than the latter; hence, it would be desirable to repeat briefly the method of preparation of an acid solution, whose strength is such that I c.c. would neutralize 0.001 gram of lime (CaO), from normal sulphuric acid solution. As the chemical equation

tion of sodium hydroxide.

Sulphuric acid Lime Calcium sulphate Water 
$$H_2SO_4$$
 +  $CaO$  =  $CaSO_4$  +  $H_2O$   $98$   $56$ 

shows, 98 grams of sulphuric acid neutralize 56 grams of lime or 49 grams of the former neutralize 28 grams of the latter.

1000 c.c.of normal sulphuric acid, which contains 49 grams of pure sulphuric acid, neutralize, like normal hydrochloric acid, 28 grams of lime. As 1000 c.c. of the standard acid solution we seek neutralize I gram of lime, or I c.c. thereof corresponds to 0.001 gram of lime, the strength of normal sulphuric acid solution is 28 times as great as that of the solution we seek, therefore, 1000 c.c. of normal sulphuric acid should be diluted with 27000 c.c. of distilled water in order to obtain an acid solution, I c.c. of which would correspond to 0.001 gram of lime. To obtain a small quantity

of such acid solution,  $\frac{1}{0.028} = 35.7$  c.c. of normal sulphuric acid

solution should be diluted to 1000 c.c. with water, for reasons similar to those explained under Standard Hydrochloric Acid Solution. This solution may be called *Twenty-eighth Normal* 

$$\left(\frac{N}{28}\right)$$
 Sulphuric Acid Solution.

Standard Acid Solution Used for Examining Samples Containing Large Quantity of Lime.—For examination of samples containing large quantity of lime, such as saccharate of lime or lime powder, it is desirable to use an acid solution whose strength is ten times as great as that of the solution just prepared. Such a solution is prepared by diluting 357 c.c. of normal sulphuric or hydrochloric acid solution to 1000 c.c. with distilled water.

Sulphur.—Symbol S; Atomic Weight 32; Valence 2, 4 and 6. This non-metallic element is found native in many volcanic regions. It occurs in several allotropic forms; namely, crystalline, amorphous, and plastic. It is a brittle solid of lemon-yellow color and is tasteless, inodorous, and insoluble in water, but soluble in carbon disulphide. It burns in air or oxygen with a clear blue flame, the product being sulphur dioxide (SO<sub>2</sub>). There are two oxides of sulphur, namely, sulphurous anhydride or sulphur dioxide and sulphuric anhydride or sulphur trioxide.

Sulphur Dioxide.—Formula SO<sub>2</sub>; Molecular weight 64. Sulphur dioxide is a gas with a suffocating odor. It is produced

by burning sulphur in air or oxygen. The reaction may be represented by the equation:

It neither supports combustion nor sustains animal or plant life. This gas dissolves slightly in water, forming a very unstable acid called sulphurous acid or hydrogen sulphite (H<sub>2</sub>SO<sub>3</sub>), the reaction being:

Sulphur dioxide Water Sulphurous acid 
$$SO_2 + H_2O = H_2SO_3$$
.

Sulphur dioxide has a great affinity for another atom of oxygen in order to form sulphuric anhydride or sulphur trioxide (SO<sub>•</sub>). When sulphur dioxide comes in contact with any substance that gives up oxygen readily, the former deprives the latter of oxygen; sulphur dioxide coming in contact with nitric for example. acid deprives it of oxygen. (This will be discussed further under SULPHURIC ACID.) It is on this account that sulphur dioxide is called a Reducing Agent. Sulphur dioxide is used as a bleaching agent, since it bleaches organic coloring matter. Its bleaching power is due to its reducing action. Organic coloring matter usually consists of carbon, hydrogen, oxygen, and nitrogen or sulphur. When sulphur dioxide deprives it of oxygen, the original composition is destroyed or the substance is converted into some simpler compounds which have no color; hence, the color is bleached.

Sulphur dioxide, it is claimed, improves the color of the beet juice, removes organic nitrogen compounds from the beet juice, and also precipitates not only free lime but that remaining dissolved in the juice in the form of some organic salts, which the carbon dioxide gas failed to separate. The fact that sulphur dioxide possesses these properties is taken advantage of in its application for the purification of beet juice.

Sulphuric Anhydride or Sulphur Trioxide.—Formula SO<sub>3</sub>; Molecular Weight 80. Sulphur dioxide, as stated previously, may be readily oxidized, forming sulphur trioxide. Sulphuric anhydride may be prepared by passing sulphur dioxide and oxygen together over finely divided platinum at a somewhat elevated temperature, the reaction being:

Sulphur dioxide Oxygen Sulphur trioxide 
$$SO_2$$
 + O =  $SO_8$ .

Sulphur trioxide is a white crystalline solid which melts at 14.8° C and boils at 46°C. It dissolves in water with great energy forming sulphuric acid:

Sulphur trioxide Water Sulphuric acid 
$$SO_3$$
 +  $H_2O$  =  $H_2SO_4$ .

Weight 98. The usual method of preparation of sulphuric acid is oxidation of sulphur dioxide by the action of nitric acid in the presence of steam in a large lead chamber, the reaction being:

Sulphur dioxide Nitric acid Water Sulphur ic acid Nitrogen trioxide 
$$2SO_2 + 2HNO_3 + H_2O = 2H_2SO_4 + N_2O_3$$
.

Nitrogen trioxide once formed acts indefinitely as a carrier of oxygen from air to sulphur dioxide in order to form sulphuric acid. Details of this reaction may be found in any text book on chemistry.

Sulphuric acid is an oily liquid and is colorless when pure. When cooled, it forms crystals. It dissolves most metals, forming sulphates of metals and liberating hydrogen gas. For example, iron dissolves in sulphuric acid, forming ferrous sulphate and hydrogen according to the equation:

Sulphuric acid Iron Ferrous sulphate Hydrogen 
$$H_2SO_4$$
 + Fe = FeSO<sub>4</sub> +  $H_2$ .

It also combines with oxides and hydroxides of alkaline metals (sodium and potassium), alkaline earth metals (calcium, strontium and barium), and heavy metals (iron, aluminum, copper, and so forth). For example, calcium oxide (lime) combines with sulphuric acid, forming calcium sulphate and water:

Calcium oxide Sulphuric acid Calcium sulphate Water
$$CaO + H_2SO_4 = CaSO_4 + H_2O.$$

Sodium hydroxide combines with sulphuric acid, forming a neutral salt of sodium sulphate and water:

Sodium hydroxide Sulphuric acid Sodium sulphate Water 
$$2NaOH + H_2SO_4 = Na_2SO_4 + 2H_2O.$$

Calcium oxide and sodium hydroxide are alkalies and may be neutralized by sulphuric acid. A definite quantity of sulphuric acid neutralizes a definite quantity of alkalies; hence, sulphuric acid of known strength may be used for the estimation of alkalies. The definition of normal sulphuric acid solution and the method for preparation thereof have been discussed under NORMAL SOLUTIONS.

When sulphuric acid is added to a solution of any soluble barium compound, barium sulphate (BaSO<sub>4</sub>) is formed, which is insoluble in water and acids. This compound will be further discussed under BARIUM.

Phosphorus.—Symbol P; Atomic Weight 31; Valence 3 and 5. Phosphorus occurs in nature in the form of phosphates, such as phosphate rocks and animal bones, both of which consist essentially of calcium phosphate, also in proteid substances in plant and animal tissues. The element is prepared by fusing bone-ash, which was previously treated with sulphuric acid, with charcoal in a porcelain retort from which phosphorus will vaporize and be collected under water. Phosphorus burns in air with a white fume. It also combines with oxygen in air, slowly under ordinary temperature, issuing a slight white fume which is phosphorescent in dark. The product in both cases is phosphorus pentaoxide  $(P_2O_5)$ , which readily dissolves in water, forming meta-phosphoric acid  $(HPO_3)$ , the reaction being:

Phosphorus pentaoxide Water Meta-phosphoric acid 
$$P_2O_5$$
 +  $H_2O$  =  $2HPO_3$ .

When boiled with water, meta-phosphoric acid is converted into ortho-phosphoric acid with the following reaction:

Ortho-phosphoric acid combines with bases in various forms. The most important ones are disodium phosphate (Na<sub>2</sub>HPO<sub>4</sub>), nor-

mal sodium phosphate (Na<sub>3</sub>PO<sub>4</sub>), acid calcium phosphate (CaHPO<sub>4</sub>), and neutral calcium phosphate (Ca<sub>3</sub> (PO<sub>4</sub>)<sub>2</sub>). Dissodium phosphate is an important reagent in chemical analysis. Neutral calcium phosphate occurs in nature as phosphate rock and bone-ash. It is treated with sulphuric acid, changed to acid calcium phosphate and used as a fertilizer in large quantities, since phosphoric acid is one of the most important of plantfoods.

Acid calcium phosphate has been proposed for purification of beet juice but has not as yet come into general use. Calcium phosphate is less soluble in sugar solution than calcium carbonate.

Any soluble magnesium compound precipitates phosphoric acid from any soluble phosphates in ammoniacal solution, forming magnesium ammonium phosphate, which will be discussed further under Magnesium. Ammonium molybdate  $((H_4N)_2MO_4)$  precipitates phosphoric acid from nitric acid solution as ammonium phospho-molybdate  $((H_4N)_3PO_4.12MO_3)$ , which is crystalline, yellow, and insoluble in nitric acid and water but soluble in ammonia, forming ammonium phosphate  $((H_4N)_3PO_4)$  and ammonium molybdate  $((H_4N)_2MO_4)$ .

Silicon.—Symbol Si; Atomic Weight 28.4; Valence 4. Silicon occurs in nature very widely distributed in combination with oxygen. With the exception of oxygen, it is the most abundant of all the elements constituting the earth. The element may be obtained by heating silicon dioxide with potassium or magnesium. It is amorphous or crystalline. Amorphous silicon is devoid of luster, inflammable when heated in air, and insoluble in water and all acids except hydrofluoric acid. Crystalline silicon forms dark lustrous octahedra hard enough to scratch glass and is infusible.

Silicon Dioxide or Silica.—Silica occurs in nature in the form of quartz or rock crystal, sandstone, flint, opal, agate, and so forth. There are silicates of various metals; the most commonly known silicates are clay and slate, both of which are aluminum silicate. Silica occurring in nature in the pure state

is quartz, which crystallizes mostly in hexagonal prisms. It is very hard and infusible in a blow-pipe flame. It has a remarkable optical property which will be discussed under the subject of the POLARISCOPE.

Pure silica may be prepared by fusing any compound containing silica with sodium carbonate, whereupon sodium will combine with silica, forming sodium silicate (Na,SiO,), which readily dissolves in hot water. When hydrochloric acid is added to the solution of sodium silicate, silicic acid (H<sub>2</sub>SiO<sub>3</sub>) and sodium chloride (NaCl) are formed. Silicic acid, when dried and heated at 100° C, decomposes into silica and water. Silica, thus precipitated, is an amorphous powder, insoluble in water and all acids excepting hydrofluoric acid; hence, pure silica may be obtained by thoroughly washing the precipitate prepared in the manner described above. A small amount of silica is usually found in spring water owing to the solubility of some of the silicates. It is also found in a small quantity in limestone in the form of sand or clay. The method for estimation of this substance will be described under LIMESTONE ANALYSIS.

The presence of a large quantity of silica in limestone is very objectionable for sugar house use. The presence of a large quantity of silica in lime for defecation of beet-juice renders the filter press cake hard and makes it very difficult to be washed. Another objection to the presence of a large quantity of silica in limestone is that it tends to fuse if overheated and adhere to the walls of the kiln, forming a scaffold or bridge which will prevent the downward progress of limestone and coke. Hence, limestone for sugar house use should be carefully examined by chemical analysis and only such limestone as contains the smallest possible amount of silica should be selected. The content of silica in good limestone does not exceed 2 per cent.

## Chapter III.

## METALLIC ELEMENTS.

Introductory.—Sodium, potassium, lithium, calcium, strontium, barium, magnesium, aluminum, chromium, iron, nickel, cobalt, manganese, zinc, silver, mercury, lead, copper, cadmium, bismuth, arsenic, antimony, tin, gold, and platinum are commonly known as metals. Sodium, potassium, and lithium are called alkaline metals; calcium, strontium, barium, and magnesium, alkaline earth metals; the remainder, heavy metals. Of these, only those metals which we have to deal with in the process of sugar manufacture will be discussed in this work.

Sodium.—Symbol Na; Atomic weight 23; Valence I. Sodium is widely distributed in nature, occurring as chloride, sulphate, carbonate, and so forth. Metallic sodium is prepared by heating a mixture of 30 parts of dry sodium carbonate, 13 parts of coal, and 3 parts of chalk in an iron cylinder from which pure metallic sodium distils over. Sodium combines with oxygen in air very readily, forming sodium oxide (Na<sub>2</sub>O). It decomposes water, forming sodium hydroxide and liberating hydrogen:

Sodium		Water		Sodium hydroxide		Hydroge	n
Na	+	$H_2O$	=	NaOH	+	H.	
dium ox	ide diss	solves in o	r combin	es with	water.	forming	so-

Sodium oxide dissolves in or combines with water, forming sodium hydroxide:

Sodium oxide		Water		Sodium hydroxide
Na <sub>2</sub> O	+	H <sub>2</sub> O	=	2NaOH.

Sodium Hydroxide.—Formula NaOH; Molecular weight 40. Sodium hydroxide is one of the strongest alkalies. It turns red litmus paper blue, phenolphthalein solution pink. Its application in the estimation of acids and the method for preparation of normal sodium hydroxide solution have been discussed under NORMAL SOLUTIONS.

Sodium hydroxide combines with carbon dioxide gas very

readily, forming sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) which readily dissolves in water, the reaction being:

This fact is taken advantage of in kiln gas analysis. The method for testing kiln gas for carbon dioxide will be described later.

Sodium Carbonate crystallizes from water solution with 10 molecules of water as water of crystallization and is known in commerce as sal-soda. If we pass an excess of carbon dioxide into a water solution of sodium carbonate, Sodium Bicarbonate (NaHCO<sub>3</sub>) is formed according to the following reaction:

When ignited, sodium bicarbonate loses carbon dioxide and water, forming normal sodium carbonate again.

Sodium hydroxide dissolves calcium salts of organic acids, forming sodium salts thereof. Sodium hydroxide combines with fat, forming soap. The process of transformation of fat into soap is called Saponification. Fat is insoluble, but soap is soluble in water. The reason sodium hydroxide is used for removing grease, is simply to saponify fatty matter in order to render it soluble in water. Evaporators and vacuum pans in sugar factories become coated with not only calcium salts of organic acids but fatty matter, as tallow is frequently used in order to prevent the juice from foaming. It is for this reason that such apparatus should be boiled occasionally with crude sodium hydroxide, or caustic soda.

Potassium.—Symbol K; Atomic weight 39; Valence 1. The properties of potassium are very similar to those of sodium. The actions of compounds of these two metals are also quite similar. Oxide, hydroxide, and carbonate of potassium need not be discussed here for they behave almost exactly the same as those of sodium.

Potassium Platinum Chloride or Potassium Chlorplatinate.— Formula K<sub>2</sub>PtCl<sub>8</sub>; Molecular weight 486. Potassium platinum chloride is a very interesting compound from the analytical standpoint. It is soluble in water but insoluble in 75 per cent alcohol, while sodium platinum chloride is soluble in either sol-This fact is taken advantage of in separating potassium from sodium. Potassium platinum chloride may be formed and separated from sodium platinum chloride by adding platinum chloride (PtCl<sub>4</sub>) to a solution of a mixture of chlorides of potassium and sodium, evaporating the solution to a syrupy condition at which potassium platinum chloride begins to precipitate in crystalline form and then adding 80 per cent alcohol to complete the precipitation. Potassium is one of the most important of all plant foods and hence an estimation thereof in soil and fertilizers is very valuable. The method for determination of potassium will be described under WATER ANALYSIS.

Potassium Cyanide.—Formula KCN; Molecular weight 65. Potassium cyanide is also an interesting compound from the analytical standpoint, as it may be used for estimation of copper. It is made by heating potassium ferrocyanide  $(K_4Fe(CN)_6)$ , which breaks up according to the following equation:

Potassium ferrocyanide		Potassium cyanide		Ferrous carbide		Nitrogen
$K_4$ Fe(CN) <sub>6</sub>	=	4KCN	+	FeC.	+	N.,

Potassium cyanide is a powerful poison and should be handled with great care. Its reaction with copper and method of estimation of copper by means of this compound will be described under COPPER.

Calcium.—Symbol Ca; Atomic weight 40; Valence 2. Calcium is widely distributed in nature and occurs mostly in combination with carbon dioxide and sulphuric acid. It is the most common of all alkaline earth metals. Limestone, calcite, marble, chalk, and Iceland spar are all carbonate of calcium (CaCO<sub>3</sub>) with more or less impurities. Gypsum and anhydrite are sulphate of calcium.

Metallic calcium is prepared by heating calcium chloride with metallic sodium or potassium. The metal is soft, malleable, brassyellow colored. It becomes very readily oxidized in air, forming calcium oxide or lime (CaO). It decomposes water, forming calcium hydroxide and liberating hydrogen:

The element combines with all inorganic and organic acids, forming acid or neutral salts of calcium, as, for example, calcium carbonate  $(CaCO_3)$ , acid calcium carbonate  $(CaH_2(CO_3)_2)$ , calcium phosphate  $(Ca_3(PO_4)_2)$ , acid calcium phosphate  $(CaHPO_4)$ , and calcium oxalate  $(CaC_2O_4)$ .

Calcium Carbonate.—Formula CaCO<sub>3</sub>; Molecular weight 100. Of all the calcium compounds, calcium carbonate is the most important from the industrial standpoint, therefore the various forms of occurrence and their properties will be discussed here.

Calcium carbonate decomposes into calcium oxide and carbon dioxide on ignition. The process of making lime based on this principal will be described later under Lime.

Calcium carbonate is insoluble in water but soluble in water containing carbon dioxide, forming acid calcium carbonate (CaH<sub>2</sub>(CO<sub>3</sub>)<sub>2</sub>), which decomposes into calcium carbonate, water, and carbon dioxide again when boiled. Spring-water and seawater contain acid calcium carbonate, which can be removed by boiling. This explains the formation of deposits of calcium carbonate in a vessel used for boiling hard water. Acid calcium carbonate may also be precipitated by neutralizing with any alkali; for example, sodium hydroxide or lime will precipitate calcium carbonate from water with the following reactions:

Acid calcium carbonate	Sodium hydroxide	Calcium carbonate	Sodium carbonate	Water
$CaH_2(CO_3)_2 +$	2NaOH =	CaCO <sub>3</sub>	+ Na <sub>2</sub> CO <sub>3</sub> +	2H <sub>2</sub> O;
Acid calcium carbonate	Lime	. '	Calcium carbonate	Water
$CaH_2(CO_3)_2$	+ CaO	==	2CaCO <sub>8</sub> +	H <sub>2</sub> O.

Marble and Calc Spar.—As is well known to the students of geology, there is a class of rocks called metamorphic rocks. The name implies that they were altered from fused mass of

primitive rocks to crystals or crystalline mass by the influence of heat and pressure during the formation of the earth's crust. During crystallization, most substances eliminate foreign substances; hence, compositions of metamorphic rocks are usually purer than those of primitive rocks. Marble and calcite or calc spar belong to this class. Of all varieties of calcium carbonates occurring in nature, Iceland spar is the purest; its crystal form is rhombohedral and it has a remarkable physical property towards light, which will be discussed in the chapter on the POLARISCOPE.

Limestone and Chalk.—The patient study of geologists reveals that the chief materials of which limestone consists are: I, shells of mollusks; 2, corals; 3, crinoids; 4, rhizopods; 5, some marine plants. Chalk is one soft variety of limestone and is mainly composed of rhizopods. It is noteworthy that all the materials composing limestone are remains of marine life.

The Theory of the Origin of Limestone may be briefly stated as follows: Calcium carbonate exists almost everywhere in the ground; coming in contact with water containing carbon dioxide, it dissolves, forming acid calcium carbonate (CaH<sub>2</sub>(CO<sub>3</sub>)<sub>2</sub>) which is carried by streams and rivers into the ocean where mollusks utilize it for their shells and corals for their reefs. the geological age when the temperature was more favorable to a low type of animal life, these animals flourished in great abundance and after fulfilling the purpose of life, the remains were deposited at the bottom of the sea. This process has been carried on for thousands of years, forming enormous layers of limestone. The pressure of water, sand and clay piled upon these beds, compressed them to hard rocks. An upheaval of the rock beds, caused by the great movements of the earth's crust, brought the limestone rocks to where we find them today. Limestone formation by marine life is still going on today at the bottom of the sea, though on a smaller scale.

The Uses of Limestone are very varied. Common limestone is used for building foundations. A good variety is polished and used as marble for building houses and monuments. Impure lime-

stone is used for making hydraulic lime, that is, cement, which sets under water; such a rock is called hydraulic lime. A great quantity of limestone rock is used for making quicklime. Lime is very useful in a great variety of industries. In sugar manufacture, lime is the most important reagent in defectation of beet juice; hence, it is imperative, for the management of a sugarhouse, to select limestone of high purity, as impure limestone is apt to introduce impurities into the beet-juice. The Nebraska sugar factories are very fortunate in having limestone rock of high quality near at hand; for example, at Weeping Water and the vicinity. A sample from that locality was analyzed with the following results:

	Per cent.
Moisture	Trace
Acid Insoluble Matter	· 1.96
Organic Matter	. Trace
Alumina	• •33
Iron Oxide ·····	• .47
Calcium Carbonate	. 96.53
Magnesium Carbonate	38
Phosphoric Acid	
Soda and Potash	
Sulphuric Acid	· Trace
•	99.67.

Calcium Oxide or Lime.—Formula CaO; Molecular weight 56. Calcium oxide is a white powder, infusible, phosphorescent at white heat, and strongly alkaline. It combines with water with the evolution of great heat, forming calcium hydroxide, commonly called water-slaked lime, which is slightly soluble in water. The reaction is represented by the following equation:

Calcium		Water		Calcium
oxide				hydroxide
CaO	+	$H_2O$	=	Ca(OH)2.

Lime readily absorbs carbon dioxide from the air, forming calcium carbonate, commonly called air-slaked lime:

Calcium oxide			Carbon lioxide	
CaO	+	CO,	=	CaCO <sub>3</sub> .

Calcium hydroxide also absorbs carbon dioxide from the air, forming calcium carbonate:

The latter fact is taken advantage of in preparing mortar, which is simply a water-slaked lime mixed with sand. The water-slaked lime takes up carbon dioxide from the air and hardens itself, forming artificial limestone.

Calcium oxide forms three compounds with sugar  $(C_{12}H_{22}O_{11})$ :

Mono-calcium Saccharate 
$$(C_{12}H_{22}O_{11}CaO)$$
  
Di-calcium Saccharate  $(C_{12}H_{22}O_{11}(CaO)_2)$   
Tri-calcium Saccharate  $(C_{12}H_{22}O_{11}(CaO)_3)$ .

The properties of these compounds will be discussed under CANE-SUGAR.

Calcium oxide neutralizes all acids, mineral or organic, forming neutral salts. Most of the calcium salts of mineral acids are more or less soluble in water, while most of the calcium salts of organic acids are insoluble in water and alkaline solutions.

Sugar beets contain, besides sugar, a large number of mineral and organic compounds, which will be discussed under Constituents of Non-sugars, somewhat in detail. Only a few of these compounds will be mentioned here in order to illustrate the value of lime in defecation of beet-juice. Two of the well known organic acids found in sugar-beets are oxalic acid (C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>=

are soluble in water. In these acids, the hydrogen atoms marked out by dotted lines are combined as base and may be replaced by calcium, forming neutral salts of calcium:

Many other organic acids contained in beets react with lime in a similar manner and are rendered insoluble in water or alkaline solution. Lime, moreover, decomposes a greater part of albumenoid or amido compounds, forming ammonia. It is also said that, when beet juice is boiled with lime, invert sugar and other starchy and gummy matter contained therein are decomposed, producing some simpler organic compounds, most of which are precipitated by lime or rendered less objectionable to crystallization of sugar even if they remain in solution. It is evident, then, that lime is a very important reagent for defecation of beet juice.

The Process of Making Lime.—As was stated previously, calcium carbonate, when strongly heated, loses carbon dioxide and is converted into lime:

Calcium carbonate		Calcium oxide		Carbon dioxide
CaCO <sub>3</sub>	=	CaO	+	CO <sub>2</sub> .

The process of making lime is based on this principle. On a large scale, lime is made by burning limestone, which is mainly composed of calcium carbonate, in a furnace called a *Lime-Kiln*. The usual dimension of lime-kiln for a sugar factory, whose capacity is five hundred tons of beets per day, is given in Fig. 1.

A lime-kiln is a tall truncated cone, made of 3/16 inch sheetiron, lined with fire clay bricks about 14 inches thick. At the base is attached another short, inverted truncated cone of 5/8 inch sheet-iron, also lined with fire clay bricks about 14 inches thick. As the diagram shows, the kiln is provided with an elevator, usually run by an electric motor, for carrying limestone and coke up to the top of the kiln, where it is fed with these materials. In starting a lime-kiln, first fill the space between the lower opening of the kiln and the ground floor with limestone rocks for a foundation and then fill the kiln with some shavings, about two tons of wood and two and one-half tons of coke. After the preliminary is completed, throw in limestone and coke alternately, keeping the proportion of coke to limestone at 1 to 10, that is, throw in 100 pounds of coke to every

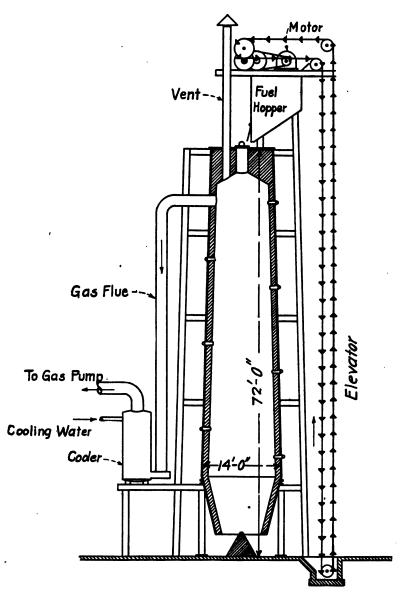


Fig. 1.—Lime-kiln for a sugar house of 500 ton per day capacity.

1000 pounds of limestone. When about 80 tons of rock are put in, start a fire at the bottom of the kiln by lighting the shavings and wood and leaving the damper at the top of the kiln open in order to cause a draught. As the wood and coke burn and the rock and coke settle down, feed the kiln with some more rock and coke, keeping it nearly three-quarters full most of the time. After about seventy hours, remove the rocks used for the foundation, and draw the first crop of lime from the lower opening of the kiln. After the kiln is well started, burned rock may be withdrawn at regular intervals. The kiln must be fed with a new supply of coke and rock after each drawing. As soon as the juice is ready to be saturated, the damper is closed and the gas pump is set in action, whereupon the gas will be drawn through the gas washer, as shown in Fig. 1, creating a draught in the kiln.

weight 128. Calcium oxalate is an important compound analytical standpoint. In estimating quantitatively, it is usually precipitated as oxalate. formed by adding oxalic acid and ammonia or ammonium oxalate to an ammoniacal solution of any calcium com-For example, if we add ammonium oxalate to ammoniacal solution of calcium chloride, calcium oxalate and ammonium chloride are formed according to the following equation:

Calcium oxalate is insoluble but ammonium chloride is soluble in water, hence calcium oxalate may be completely separated by filtering and washing. Calcium oxalate, when ignited, is converted into calcium carbonate at low heat and into calcium oxide at bright red heat:

Calcium oxalate		Calcium carbonate		Carbon monoxide
CaC <sub>2</sub> O <sub>4</sub>	=	CaCO <sub>3</sub>	+	co;
Calcium carbonate		Calcium oxide		Carbon dioxide
CaCO <sub>3</sub>	===	CaO	+	CO

The method of estimation of calcium as oxide is based on this principle, and the details of the determination will be described under LIMESTONE ANALYSIS.

Barium and Strontium.—The properties of these elements and their compounds will not be described here as they are not of special interest to sugar manufacturers. Oxides of these elements have been proposed for defecation of beet juice but they have not come to general use as they are more expensive than lime.

Barium Sulphate.—Formula BaSO<sub>4</sub>; Molecular weight 232. Barium sulphate is an important compound from the analytical standpoint. It is formed by adding barium chloride to a solution of any soluble sulphate. For example, if we add barium chloride to a solution of sodium sulphate, barium sulphate and sodium chloride are formed, according to the following equation:

Barium sulphate is insoluble in water or acid either cold or hot, while sodium chloride is readily soluble in water, hence the former may be completely separated from the latter by filtering and washing. Ignition does not fuse or decompose barium sulphate. For this reason, sulphuric acid in all soluble sulphates is estimated as barium sulphate. The method for determination will be described under LIMESTONE ANALYSIS.

Magnesium.—Symbol Mg; Atomic Weight 24; Valence 2. Magnesium is found in nature in the forms of chloride (carnallite, MgKCl<sub>3</sub>+6H<sub>2</sub>O), sulphate (epsom salt, MgSO<sub>4</sub>), carbonate (magnesite, MgCO<sub>3</sub>), silicate (soap stone, serpentine, MgSiO<sub>4</sub>), and so forth. The metallic element is obtained by electrolyzing carnallite, that is, heating the substance in a clay crucible while one pole of a battery is dipped in the fused mass and the other connected with the crucible. The metal is silver white, soft, malleable, and burns with a brilliant flame, forming magnesium oxide or magnesia. In common practice, magnesia is obtained by strongly heating magnesium carbonate. Magnesia is insoluble in water but soluble in acids forming corresponding

salts. Magnesium sulphate is readily soluble in water. Magnesium carbonate is insoluble in pure water but soluble in water containing carbon dioxide. It is for this reason that springwater almost always contains more or less magnesium compounds. Water containing a large quantity of magnesium compounds is unfit for drinking as it acts as a purgative. Such water is also objectionable for sugar house use as it introduces some impurities into the beet juice during the diffusion process and helps to increase the production of molasses. All soluble magnesium compounds are melasgenic, that is, they are molasses producing substances.

Magnesium Ammonium Phosphate is an important compound from the analytical standpoint. It is formed by adding disodium phosphate (Na<sub>2</sub>HPO<sub>4</sub>) to any solution of magnesium compounds containing ammonia. For instance, if we add disodium phosphate to a solution of magnesium sulphate containing ammonia, magnesium ammonium phosphate, sodium sulphate, and water are formed:

Magnesium Disodium Sodium Magnesium ammonium Ammonia Water sulphate phosphate phosphate sulphate  $MgSO_4 + Na_2HPO_4 + H_4NOH = MgH_4NPO_4 + Na_2SO_4 + H_2O.$ Magnesium ammonium phosphate is insoluble in water, especially in ammoniacal solution, while sodium sulphate is readily soluble in water, therefore the former may be separated completely from the latter by filtering off and washing the precipitate. When ignited, magnesium ammonium phosphate is converted into magnesium pyrophosphate, ammonia being liberated:

Magnesium ammonium Magnesium phosphate pyrophosphate Ammonia Water  $2 MgH_4NPO_4 = Mg_2P_2O_7 + 2 H_3N + H_2O$ .

The method for determination of magnesium is based on this principle and will be described under LIMESTONE ANALYSIS.

Heavy Metals.—The very commonly known heavy metals are silver, mercury, lead, copper, bismuth, tin, arsenic, antimony, cobalt, chromium, nickel, manganese, zinc, iron, and aluminum. Of these, only copper, silver, lead, iron, and aluminum need be discussed here, as the knowledge of the properties of these ele-

ments and their compounds aids sugar house chemists in their work.

Iron.—Symbol Fe; Atomic weight 56; Valence 2 and 3. Iron occurs in nature in the forms of oxides (magnetite, Fe<sub>2</sub>O<sub>4</sub>, hematite, Fe<sub>2</sub>O<sub>3</sub>, and limonite), sulphides (ironpyrites, FeS<sub>2</sub>), carbonate (siderite, FeCO<sub>3</sub>), and so forth.

The theory of the formation of iron ore may be stated as follows: During the geological age, water containing carbon dioxide or organic acids dissolved iron oxide, which exists almost everywhere in the ground, and carried it through streams into low places or marshes where, in course of time, the water evaporated and the iron ore was deposited. This process probably continued thousands of years, accumulating enormous quantities of iron ore.

Metallic Iron may be obtained by reducing any oxide of iron with carbonaceous matter such as coal, coke or charcoal in a furnace into which a blast of hot gas is forced. In this process, iron oxide gives up its oxygen to carbon, forming carbon dioxide or carbon monoxide, transforming itself into a metallic condition. A varied quantity of flux of sand and lime is used to protect reduced iron from oxidation. Molten iron is withdrawn from an opening at the bottom of the furnace and casted in sand moulds. This is called Cast-Iron. Cast-iron contains a considerable quantity of foreign matter, such as carbon, silicon, sulphur, and phosphorus. It is light gray in color, brittle, and comparatively easily fusible.

In order to obtain iron of greater tensile strength, the castiron or "pig-iron" is remelted in a chamber called a puddling furnace, where carbon and sulphur are oxidized to carbon dioxide and sulphur dioxide, respectively, which escape as gases and silicon and phosphorus are oxidized and separated as slag. The iron thus freed from a large part of foreign matter becomes tough and malleable and its melting point is raised. Such iron is called Wrought-Iron.

When a content of carbon in iron is increased within a certain limit, iron becomes harder and more elastic. This effect is accomplished in the process known as the Bessemer Process. In

this process, a strong blast of air is blown through a molten mass of cast-iron, by means of which a large part of the impurities is oxidized and removed, and to this mass a certain amount of molten cast-iron, whose carbon content is known, is added and thoroughly mixed, so that a desired amount of carbon may be distributed throughout the entire mass. The product is called *Steel*. The hardness and elasticity vary according to the quantity of carbon present.

The chemical composition of cast-iron, wrought-iron, and steel varies considerably but may be roughly stated as follows:

	Iron	Carbon	Silicon	Sulphur	Phos- phorus	Man- ganese	Sp.	Weight per cubic inch in pounds
Cast-Iron	92.370	3.650	2.440	.020	1.250	.280	7.15	.260
Wrought-Iron	99.573	.015	. 156	.008	.231	.017	7.80	.278
Steel	99.044	.867	.057	.018	.014	• • • •	7.84	.283.

Iron prepared from ores always contains some impurities. Chemically pure iron may be obtained by igniting pure oxide or oxalate in a current of hydrogen.

Properties of Iron and Its Compounds.—Metallic iron dissolves in many mineral acids, forming respective salts of iron. For example, hydrochloric acid dissolves metallic iron, forming ferrous chloride and hydrogen, the reaction being represented by the equation:

When oxidized, ferrous chloride is converted into ferric chloride:

Sulphuric acid also dissolves iron, forming ferrous sulphate and hydrogen:

When oxidized, ferrous sulphate is converted into ferric sulphate:

Ferrous sulphate Oxygen Sulphuric sulphate Sulphate Sulphate Water 2 FeSO<sub>4</sub> + O + 
$$H_2SO_4$$
 =  $Fe_2(SO_4)_3$  +  $H_2O$ .

It may be seen from these reactions that iron forms two series of compounds, namely, ferrous and ferric compounds.

Ferrous salts form, with any alkali, white precipitate of ferrous hydroxide (Fe(OH)<sub>2</sub>), which turns brown on standing, being oxidized to ferric hydroxide (Fe(OH)<sub>8</sub>). Ferric salts form bright brown precipitate of ferric hydroxide with any alkali; for example:

Ferric hydroxide is insoluble in water, ammoniacal solution or even in strong solution of sodium or potassium hydroxide; hence, precipitate of ferric hydroxide may be separated from other substances soluble in alkaline solution by filtering off and washing. Ferric hydroxide, when ignited, loses water, forming ferric oxide:

Ferric Ferric oxide Water 
$$2 \text{ Fe}(OH)_3 = Fe_2O_3 + 3 H_2O$$
.

Gravimetric determination of iron is based on this property of ferric oxide and the method will be described under LIMESTONE ANALYSIS.

Ferric oxide and ferric hydroxide are soluble in acid, forming ferric salts:

Solubility of Ferric Oxide and Ferric Hydroxide in Sugar Solution.—Another interesting property of ferric oxide and ferric hydroxide is their solubility in a strong sugar solution, especially in the presence of sucrate of lime, and no alkali is able to precipitate iron therefrom. When sugar crystallizes out from beet juice, it usually eliminates most of the impurities; iron, however, goes into combination with sugar, though in minute quantity, and acts as a coloring matter, causing, when present in sufficient quantity, the sugar crystals to acquire grayish yellow color. The

chief source of iron in the juice is probably limestone. Sometimes the water used in a sugar house contains some iron. Some iron is undoubtedly dissolved from tanks, pumps, pipes, evaporators, and vacuum pans, as they are usually made of iron, and may be readily oxidized by air and water and dissolved by sugar. The quantity of iron introduced from these sources is really insignificant, but it should be remembered that it does not require more than a trace of iron to cause sugar crystals to acquire color. According to the author's investigation, a fillmass or massecuite of 85 per cent purity, which yielded grayish yellow colored sugar, contained only 0.02 per cent ferric oxide.

Determination of Iron by Means of Potassium Permanganate.— Ferrous compounds become readily oxidized by any oxidizing agent; for example, potassium permanganate readily oxidizes ferrous sulphate according to the following equation:

This equation indicates that a definite quantity of potassium permanganate oxidizes a definite quantity of ferrous sulphate, therefore, if the strength of potassium permanganate solution be known, the amount of iron that has been oxidized may be calculated.

Weigh exactly 1.58 grams of chemically pure crystallized potassium permanganate, dissolve in a little water, transfer to a 500 c.c. graduated flask and make up to the mark with water. I c.c. of this solution corresponds to 0.0056 gram of iron, but it is safest to standardize the solution with metallic iron (piano wire) or ferrous ammonium sulphate (FeSO<sub>4</sub>(H<sub>4</sub>N)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O) before using. Weigh out about 1.5 grams of ferrous ammonium sulphate, dissolve in 75 c.c. of water, add 10 c.c. of dilute sulphuric acid and then add the potassium permanganate solution from a burette until a faint pink color of the liquid remains permanent. Assuming that 1.542 grams of ferrous ammonium sul-

phate, which contains exactly 1/7 its weight of iron, required 39.32 c.c. of the potassium permanganate solution, the value of 1 c.c. of the permanganate solution would be

$$\frac{1.542}{7 \times 39.32}$$
 = 0.0056 gram iron.

In estimating iron in some sample containing iron, weigh out about 2 grams of the sample, dissolve, and digest in strong hvdrochloric acid, filter off the insoluble matter and wash thorough-Add an excess of ammonia to the filtrate, whereupon all iron and alumina will be precipitated in the form of hydroxides. Collect the precipitate in a filter paper and wash thoroughly. Dissolve the precipitate in 10 c.c. of dilute sulphuric acid, wash the filter paper thoroughly, transfer the solution to a small Erlenmeyer flask, add granulated zinc free from iron, close the flask with a rubber stopper provided with a Bunsen valve, and heat the solution slowly until a drop thereof no longer gives color with potassium sulphocyanate, all the iron being reduced to ferrous sulphate. When the reduction is completed and all the zinc is dissolved, remove the stopper from the flask and add, from a burette, the potassium permanganate solution to the solution while hot until the color of the solution remains slightly pink for five minutes. Assuming that 55 c.c. of the potassium permanganate solution were required to reach the end reaction, iron contained in the sample would be

$$\frac{0.0056 \times 55 \times 100}{2} = 15.4 \text{ per cent.}$$

A Bunsen valve is merely a short small thick rubber tubing provided with a slit at the side. One end of the tube is connected with a rubber stopper by means of a short glass tubing, while the other is closed with a short solid glass rod. When a solution is boiled in a flask closed with a valve of this arrangement, the valve allows the vapor to escape but prevents the entrance of air.

Aluminum.—Symbol Al; Atomic weight 27; Valence 3. Aluminum occurs widely distributed in nature chiefly as silicate (clay), oxide (corundum, Al<sub>2</sub>O<sub>3</sub>), and fluoride (cryolite, Na.

AlFl<sub>6</sub>). The metal may be obtained by electrolizing a molten mass of cryolite and corundum in an iron pot. The pot acts as one electrode while the stick of carbon dipped in the molten mass serves as the other electrode. The metal separating from the carbon stick will collect at the bottom of the crucible and be drawn. The metal is white, soft, malleable, ductile, and highly lustrous. Its specific gravity is 2.7.

Aluminum is not of special interest to sugar manufacturers. It is, however, found in water and limestone used in sugar factories together with iron and introduced into the juice as an impurity; hence, the sugar-house chemist should be familiar with the properties of compounds of aluminum and the principle of the method for the estimation thereof.

Metallic aluminum is soluble in hot but not cold sulphuric acid or nitric acid but readily soluble in cold hydrochloric acid. All these acids form with aluminum corresponding salts: Aluminum sulphate  $(Al_2(SO_4)_3)$ , aluminum nitrate  $(Al(NO_3)_3)$ , and aluminum chloride  $(AlCl_3)$ . When ammonium sulphate  $((H_4N)_2SO_4)$  is added to aluminum sulphate, a double compound, aluminum ammonium sulphate  $(AlH_4N(SO_4)_2+12H_2O)$  is formed. This double compound is known in commerce as alum. When any alkali solution is added to a solution of any aluminum compound, aluminum is precipitated as hydroxide; for example, when ammonia is added to a solution of aluminum sulphate, aluminum hydroxide and ammonium sulphate are formed:

Aluminum sulphate Al
$$_2(SO_4)_8$$
 +  $6H_4NOH$  =  $2Al(OH)_3$  +  $3(H_4N)_2SO_4$ .

Aluminum hydroxide is insoluble in an excess of ammonia but soluble in that of potassium hydroxide, forming potassium aluminate, which is soluble in water:

When ammonium chloride is added to a solution of potassium aluminate, aluminum hydroxide may be reprecipitated with the following reactions:

Potassium aluminate Ammonium chloride Potassium aluminate Al(OK)<sub>3</sub> + 
$$3H_4NC1$$
 =  $3KC1$  +  $Al(OH_4N)_3$ ;

Ammonium aluminate Water Ammonium hydroxide Al(OH<sub>4</sub>N)<sub>3</sub> +  $3H_2O$  =  $3H_4NOH$  +  $Al(OH_3)_8$ .

From these reactions, it may be seen that when aluminum is precipitated together with iron as hydroxides by means of ammonia aluminum may be separated by dissolving aluminum hydroxide with an addition of potassium hydroxide and filtering off and washing the iron hydroxide. Aluminum hydroxide may be reprecipitated by adding ammonium chloride to the filtrate. The method for the estimation of aluminum oxide will be described under LIMESTONE ANALYSIS.

Lead.—Symbol Pb; Atomic weight 207; Valence 2. Lead occurs in nature chiefly as sulphide (galena or galenite, PbS), sulphate (anglesite, PbSO<sub>4</sub>), and carbonate (cerussite, PbCO<sub>3</sub>). Metallic lead is obtained by roasting galena, partly converting to lead oxide (PbO) and lead sulphate, and finally heating the mixture in a chamber from which the air is excluded and where the following reactions take place:

Lead oxide		Lead sulphide		Lead		Sulphur dioxide
2PbO	+	PbS	==	3Pb	+	SO <sub>2</sub> ;
Lead sulphate		Lead sulphide		. Lead		Sulphur dioxide
$PbSO_4$	+	PbS	==	2Pb	+	2SO <sub>2</sub> .

Lead is a soft, malleable, bluish gray metal. Its specific gravity is 11.4 and it melts at 325°C. It is used for a variety of purposes, such as water pipes, sulphur gas pipes, vessels used for manufacturing sulphuric acid, and as a constituent of various alloys.

There are three oxides of lead, namely, lead oxide (PbO) or litharge, lead peroxide (PbO<sub>2</sub>), and red lead or minimum (Pb<sub>3</sub>O<sub>4</sub>). Litharge is formed by heating lead in air.

Metallic lead is practically insoluble in sulphuric acid or hydrochloric acid but readily soluble in nitric acid, forming lead nitrate with the following reaction:

Nitric Lead Lead Hydrogen acid 
$$\frac{Lead}{nitrate}$$
 Hydrogen  $2HNO_3$  + Pb =  $Pb(NO_3)_2$  +  $H_2$ .

It also dissolves in acetic acid (CH<sub>8</sub>COOH), forming lead acetate (Pb(CH<sub>8</sub>COO)<sub>2</sub>), with the following reaction:

Lead oxide also dissolves in acetic acid:

Lead acetate is soluble in water. When lead acetate solution is warmed with lead oxide (litharge), a basic acetate of lead (Pb<sub>2</sub>O(CH<sub>8</sub>COO)<sub>2</sub>) is formed. This compound is also soluble in water.

There are several lead salts insoluble in water or acid. When any soluble lead salt is treated with ammonium carbonate, lead carbonate (PbCO<sub>3</sub>) is formed. When a soluble lead salt is treated with sodium carbonate, a basic carbonate of lead with the formula 3PbO2CO<sub>8</sub> + H<sub>2</sub>O is formed. These carbonates are insoluble in water but soluble in acid. Basic carbonate of lead is used for pigment and is known in commerce as "white lead." If we add sulphuric acid or any soluble sulphate to a solution of soluble salt of lead, lead sulphate is precipitated. phate is soluble in hot concentrated hydrochloric acid or ammonium acetate but insoluble in sulphuric acid, nitric acid or water; hence, in analytical work, lead is usually estimated as lead sulphate. Lead also forms, with many organic acids, compounds insoluble in water; for example, if we add a solution of lead acetate to a solution of oxalic acid or any soluble oxalate, lead oxalate is precipitated, the reaction being represented by the following equation:

Similar reactions take place between lead acetate and other organic acids. Most of the lead salts of organic acids are insoluble in water and organic acids themselves, but they are all soluble in mineral or inorganic acids.

Many organic compounds are optically active, that is, they have property similar to that of cane-sugar toward polarized light. Beet juice, as will be discussed under Constituents of Non-sugars, contains, besides sugar, many organic compounds several of which are optically active. Basic acetate or subacetate of lead precipitates most of these optically active substances and coloring matter contained in beet juice. It is on this account that subacetate of lead is used for clarifying beet juice and other sugar-house products before polarization. The reason subacetate of lead instead of other soluble lead salts is used for clarifying sugar solutions, is that lead subacetate precipitates not only all the compounds which could be precipitated but some of those which could not be removed by other soluble lead salts.

The method for preparation of lead subacetate solution used in sugar analysis will be described under General Methods for Sugar Analysis.

Copper.—Symbol Cu; Atomic weight 63.6; Valence 2. Copper occurs in nature chiefly as metal, oxide (cuprite Cu<sub>2</sub>O), sulphide (chalcopyrite, CuFeS<sub>2</sub>), and carbonate (malachite, CuCO<sub>8</sub>). Metallic copper is obtained by roasting oxide of copper with charcoal, or a mixture of oxide and sulphide of copper. The metal thus obtained, however, contains more or less impurities. It is usually refined by the electrolytic method. In this method, a thick plate of impure copper is suspended in dilute sulphuric acid contained in a tank and connected with the positive pole of a battery and a sheet of pure copper connected to the negative pole is suspended in the same tank at some distance from the impure copper plate. Pure copper dissolved from the impure plate is transferred to and deposited on the pure copper plate, while the impurities settle down to the bottom of the tank. Metallic copper is ductile and a good conductor of electricity; it melts at 1091°C; its specific gravity is 8.95. It is very extensively used for electrical apparatus, coins. various vessels, copper plating and as a constituent of various alloys such as brass, bronze, and gun-metal.

Copper is insoluble in hydrochloric acid and cold sulphuric



acid, but soluble in hot sulphuric acid, forming copper sulphate  $(CuSO_4)$  and sulphur dioxide. It readily dissolves in nitric acid, forming copper nitrate  $(Cu(NO_3)_2)$  and nitric oxide (NO).

Metallic copper may be precipitated from a solution of copper sulphate by iron, aluminum, or zinc, as, for example:

Characteristic Reactions of Copper Compounds.—Copper salts in alkaline solution may be reduced to cuprous oxide by reducing sugars, such as invert sugar, glucose, levulose, or lactose; for example, if we heat glucose with Fehling's solution, a red precipitate of cuprous oxide is formed. Fehling's solution is a mixture of copper sulphate, potassium hydroxide, and sodium

potassium tartrate ( 
$$^{\mathrm{CH-OH-COOK}}_{\mathrm{CH-OH-COONa}}$$
 ). A certain quantity

of reducing sugar reduces a certain quantity of cuprous oxide; for example, 0.56 gram of glucose reduces 1.252 grams of cuprous oxide (Cu<sub>2</sub>O) or 1 gram of metallic copper. This fact is taken advantage of in the estimation of reducing sugars. The method for the preparation of Fehling's solution and that for the estimation of reducing sugars by means of this solution will be described under ESTIMATION OF INVERT-SUGAR.

When soluble copper salts are treated with ammonia, a double compound of ammonia and copper is formed; for example, if we treat copper sulphate with ammonia, cupric ammonium sulphate  $(Cu(H_8N)_4SO_4)$  is formed. This compound gives a beautiful azure blue color to the solution. Copper chloride  $(CuCl_2)$  and copper nitrate also form similar compounds with ammonia, namely, copper ammonium chloride  $(Cu(H_8N)_4Cl_2)$  and copper ammonium nitrate  $(Cu(H_8N)_4(NO_8)_2)$ . Both compounds give a beautiful azure blue color to the solution.

Cyanides of copper are also very interesting compounds from the analytical standpoint. They are formed by treating any soluble copper salts with potassium cyanide; for example, if we treat ammoniacal solution of copper sulphate  $(Cu(H_sN)_4SO_4)$ .

H<sub>2</sub>O) with potassium cyanide, yellow cupric cyanide is first formed with the following reaction:

Copper ammonium sulphate cyanide cyanide cyanide phate water Ammonia  $2Cu(H_3N)_4SO_4.H_2O + 4KCN = 2Cu(CN)_1 + 2K_2SO_4+H_2O + 8H_3N$ . The cupric cyanide thus formed, however, is immediately converted into white cuprous cyanide, which readily dissolves in an excess of potassium cyanide, the reaction being:

The liberated cyanogen combines with ammonia forming ammonium cyanide (H<sub>4</sub>NCN). When all the copper is converted into cuprous cyanide, the characteristic blue color disappears entirely.

The reactions just cited may be applied for analytical purposes, as a definite quantity of copper requires a definite quantity of potassium cyanide in order to cause its characteristic blue color to disappear entirely.

The Method for Estimation of Copper by Means of Potassium Cyanide.—As was stated previously, the amount of invert-sugar may be estimated from the amount of cuprous oxide it reduces; hence, it will be convenient to discuss here the method for estimation of copper so that it may be applied later for the estimation of invert-sugar. There are several methods used for the determination of copper, but the one based on the reaction between copper and potassium cyanide is the simplest, fairly accurate and sufficiently close for sugar-house work; hence, this method alone will be described in this work.

Dissolve about 20 grams of potassium cyanide in one liter of distilled water and standardize the solution with pure metallic copper. In standardizing, weigh about 0.15 gram of pure copper foil polished with fine sand, as accurately as possible, dissolve it in about 5 c.c. of dilute nitric acid in a small porcelain evaporating dish and add ammonia in a slight excess. Now fill a 50 c.c. burette with the potassium cyanide solution just prepared to exactly the zero mark and run the solution into the

dish containing the copper, stirring continually, until the characteristic blue color just disappears. Assume that it required 30 c.c. of the potassium cyanide solution to reach this point, then the value of I c.c. of the potassium cyanide solution in terms of copper would be  $\frac{0.15}{30} = 0.005$  gram. The purity of so-called pure copper, however, is usually 99.8 per cent; hence, the true value of I c.c. of the potassium cyanide solution would be  $0.005 \times 0.998 = 0.00499$  gram copper. Repeat this operation several times and take the average result for final calculation.

	EXAMPLE.	
Copper grams		Potassium cyanide c.c.
.1548	required	29.9
. 1486	66	29.6
.1072	"	21.0
.4106	"	80.5

The average value of 1 c.c. of the potassium cyanide solution would be  $\frac{0.4106}{80.5} = 0.0051$  gram copper and the true value would be  $0.0051 \times 0.008 = 0.0050898$  gram copper. Potassium cyanide solution thus standardized may be used for the estimation of copper contained in any compound after proper treatment.

In case copper in cuprous oxide reduced by some reducing sugar from Fehling's solution is to be estimated, filter off the oxide and wash thoroughly with hot water, dissolve in dilute nitric acid, add ammonia in an excess and the standardized potassium cyanide solution until the blue color of the solution just disappears. Note the number of c.c. of the potassium cyanide solution required. Assuming that 50 c.c. of the potassium cyanide solution are required, the metallic copper in the oxide would be  $50 \times 0.0050898 = 0.25449$  gram.

Silver.—Symbol Ag; Atomic weight 107; Valence 1. Silver occurs in nature as metal, sulphide (argentite, Ag<sub>2</sub>S), and chloride (horn silver, AgCl). Silver sulphide is usually found intimately mixed with galena. Metallic silver is commonly separated from lead by amalgamating with zinc, as lead is usually ob-

tained by roasting galena containing silver sulphide. In this process, a small amount of zinc is added to a molten mass of lead and well stirred, whereupon the silver forms an alloy with the zinc. This alloy, being of less specific gravity and having a higher melting point, rises to the surface and forms a crust on cooling slightly. The crust thus formed is skimmed off with a perforated ladle and subjected to a distillation in a graphite retort in order to remove the zinc. A small amount of lead remaining with the silver is separated by cupellation, that is, by strongly heating in order to oxidize the lead, in a vessel made of boneash and called a cupel, which absorbs oxidized lead.

Silver is very white, soft, ductile, malleable, and heavy, its specific gravity being 10.5. It is the best conductor of heat and electricity. It is alloyed with a small percentage of copper when used for coin and silver-ware, in order to increase its hardness.

Silver can not be oxidized at any temperature, but ozone forms with silver a silver oxide  $(Ag_2O_2)$ . Ordinary silver oxide  $(Ag_2O)$  is formed by treating silver nitrate with sodium or potassium hydroxide. Silver is slightly acted on by hydrochloric acid but is readily dissolved by nitric acid and hot sulphuric acid, forming silver nitrate  $(AgNO_3)$ , and silver sulphate  $(Ag_2SO_4)$ , respectively.

Silver nitrate is soluble in water and is a very important reagent in analytical work. When any soluble chloride is added to a solution of silver nitrate, silver is precipitated as chloride, which is soluble in ammonia but insoluble in water or nitric acid. Insolubility of silver chloride in water or nitric acid is taken advantage of in estimation of chlorine, since silver chloride may be filtered off, washed thoroughly, dried and weighed, and chlorine may be calculated from the weight of silver chloride. The method of estimation of chlorine based on this principle will be described under WATER ANALYSIS.

## Chapter IV.

## ORGANIC CHEMISTRY.

It would be proper to make some brief remarks on Organic Chemistry in order to show what places Cane-Sugar and the Organic Compounds occurring in beets occupy in the field of chemistry.

Hydrocarbons.—There are several series of hydrocarbons; namely, those of methane, ethylene, acetylene, and benzene. The carbon atom being quadrivalent, the molecular structure of the hydrocarbons may be explained by the formula given under structural formula:

	METHANE SERIES.	
Names	Empirical formula	Structural formula
		H
36-41	011	
Methane	CH <sub>4</sub>	н—с—н
		$^{H}$
		нн
<b>T</b> 44	0.77	-
Ethane	$C_2H_6$	H-Ç-Ç-H
		$\overset{\mathbf{h}}{\mathbf{h}}$
		нин
<b>5</b>	0.44	H-C-C-C-H
Propane	$C_3H_8$	н-с-с-с-н
		ннн
		нннн
D.,4	0.77	H-C-C-C-C-H
Butane	$C_4H_{10}$	H-C-C-C-H
		<b>####</b>
and so forth.		
	ETHYLENE SERIES.	
•	Empirical formula	Structural formula
		H-C-H
Ethylene	$C_2H_4$	·
		H—Č—H

Names	ETHYLENE SERIES—(Continued) Empirical formula	Structural formula H
Propylene	C <sub>3</sub> H <sub>6</sub>	H—C—C—H
Butylene	$\mathrm{C_4H_8}$	H—C—H H—C—C—H    H    H    H    H    H
and so forth.	ACETYLENE SERIES. Empirical formula	Structural formula
Acetylene	C <sub>2</sub> H <sub>2</sub>	С—н С—н
Allylene	C3H4	С—Н     Н С—С—Н Н
and so forth.	BENZENE SERIES. Empirical formula	Structural formula H
Benzene •	C₀H₀	H-C C-H
Toluene	$C_7H_8$	H H-C C-C-H H-C C-H

## BENZENE SERIES—(Continued).

and so forth.

As may be seen from the formula, any two consecutive members of any series differ by CH<sub>2</sub>; for example when CH<sub>2</sub> is added to CH<sub>4</sub> (methane), we obtain C<sub>2</sub>H<sub>6</sub> (ethane). This regular increase in composition is known as homology.

Hydrocarbons of the methane series are found in petroleum and those of the benzene series in coal tar.

The simplest method to prepare methane from elements is by treating aluminum carbide with water, in which the following reaction takes place:

In common practice, methane is prepared by heating a mixture of sodium acetate and sodium hydroxide:

Halogen Derivatives of Hydrocarbons.—All hydrocarbons have derivatives of halogens (chlorine, bromine and iodine), and of oxygen, nitrogen, sulphur, and phosphorus. When chlorine or bromine is brought in contact with methane or other hydrocarbons, one or more atoms of hydrogen are replaced by chlorine or bromine; for example, when methane gas and chlorine are brought together in sunlight, a compound with the formula CH<sub>2</sub>Cl is formed which is known as mono-chlor-methane. When the

compound is treated with more chlorine the following compounds are successively formed:

Di-chlor-methane	•	CH,Cl,;
Tri-chlor-methane		CHCl <sub>3</sub> ;
Tetra-chlor-methane		CC14.

Bromine forms with methane, mono-bromo-, di-bromo- and tri-bromo-methane. Iodine forms with methane, mono-iodo-, di-iodo-, and tri-iodo-methane. The well known compound, chloro-form, is tri-chlor-methane, and is commonly made by treating alcohol with bleaching powder. That well known antiseptic, iodo-form, is tri-iodo-methane (CHI $_{\rm 3}$ ) and made by treating alcohol with an alkali and iodine. All these hydrocarbons whose hydrogen atoms are replaced by chlorine, bromine, or iodine are known as substitution products.

Ethane also forms, with chlorine, bromine, and iodine, compounds similar to those of methane:

Chlor-ethane or ethyl chloride	C <sub>2</sub> H <sub>5</sub> Cl;
Bromo-ethane or ethyl bromide	C <sub>2</sub> H <sub>5</sub> Br;
Iodo-ethane or ethyl iodide	$C_2H_5I$ .

All these compounds are readily made by treating alcohol with corresponding acids, that is, hydrochloric acid, hydrobromic acid, and hydriodic acid.

These substitution products are extremely interesting compounds as many of the higher hydrocarbons have been prepared by building up by means of these compounds; for example, ethane may be built up by treating mono-iodo-methane with metallic sodium. The reaction involved therein may be expressed by the following equation:

Propane may be obtained by treating a mixture of methyl iodide and ethyl iodide with metallic sodium:

Methyl Ethyl iodide			Sodium Propane				Sodium iodide	
CH-I	+	C.H.I	+	2 Na	=	C.H.	+	2 NaI.

Oxygen Derivatives of Hydrocarbons.—The most important of the oxygen derivatives are alcohols, ethers, aldehydes, and

acids. The relations existing between hydrocarbons and their oxygen derivatives may be readily seen from the following tabulated formulas:

```
\begin{array}{cccc} \text{Hydrocarbons} & \text{Alcohols} & \text{Ethers} \\ \text{CH}_4(\text{Methane}) & \text{CH}_4\text{O}(\text{Methyl alcohol}) & (\text{CH}_5)_2\text{O}(\text{Methyl ether}) \\ \text{C}_2\text{H}_6(\text{Ethane}) & \text{C}_2\text{H}_6\text{O}(\text{Ethyl alcohol}) & (\text{C}_2\text{H}_5)_2\text{O}(\text{Ethyl ether}) \\ & & \text{Aldehydes} & & \text{Acids} \\ \text{CH}_2\text{O}(\text{Methyl aldehyde}) & \text{CH}_2\text{O}_2(\text{Formic acid}) \\ \end{array}
```

C<sub>2</sub>H<sub>4</sub>O(Ethyl aldehyde) C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>(Acetic acid). Alcohols.—All alcohols contain the hydroxyl group OH and hence may be regarded hydroxides of hydrocarbon radicals which are analogous to hydroxide of sodium or potassium. Alcohols containing one hydroxyl group like methyl alcohol (CH<sub>3</sub>OH), ethyl alcohol (C<sub>2</sub>H<sub>5</sub>OH) are called mono-acid-alcohols; those containing two hydroxyl groups like glycol (C<sub>2</sub>H<sub>4</sub>(OH)<sub>2</sub>), diacid-alcohols; those containing three hydroxyl groups like glycerine (C<sub>3</sub>H<sub>5</sub>(OH)<sub>3</sub>), tri-acid-alcohols. Groups CH<sub>8</sub>, C<sub>2</sub>H<sub>5</sub> and so forth are called Radicals.

Methyl Alcohol, CH<sub>4</sub>O=CH<sub>3</sub>OH.—Methyl alcohol is known, in commerce, as wood-alcohol or wood-spirits. It may be prepared by treating one of the mono-halogen derivatives of methane with sodium or potassium hydroxide, as, for example:

Methyl bromide		Sodium hydroxide		Methyl alcohol		Sodium bromide
CH,Br	+	NaOH	=	CH,OH	+	NaBr.

Wood-alcohol in commerce, however, is obtained by the dry distillation of wood. Wood-alcohol is usually a by-product of a charcoal factory. The distillate contains many other substances. Pure methyl alcohol may be separated by fractional distillation as it boils at 66.7° C. It burns with a non-luminous flame. It is an excellent solvent for fats, oils, resins, and other organic substances.

Ethyl Alcohol, C<sub>2</sub>H<sub>6</sub>O=C<sub>2</sub>H<sub>5</sub>OH.—Ethyl alcohol is commonly known as alcohol or spirits of wine. It may be prepared, like methyl alcohol, by treating one of the mono-halogen derivatives of ethane with sodium hydroxide:

In commercial practice, alcohol is obtained by subjecting a solution containing glucose or fructose to fermentation in which these sugars are broken up into alcohol and carbon dioxide by the action of minute fungi known as yeast plants or saccharomyces cerevisiae. The reaction may be represented by the following equation:

Alcohol is separated from the fermented liquid by distillation, as its boiling point is lower than that of water. Alcohol thus obtained contains from 4 to 5 per cent of water. In order to obtain alcohol free from water, or absolute alcohol, it must be redistilled with some dehydrating agent, such as quicklime or anhydrous copper sulphate. Absolute alcohol boils at 78.3°C and solidifies at —130.5°C. Alcohol burns with a non-luminous flame and is one of the best solvents for organic substances.

Alcohol combines with sulphuric acid, forming ethyl sulphuric acid:

Alcohol Sulphuric acid Ethyl sulphuric acid Water 
$$C_2H_5OH$$
 +  $H_2SO_4$  =  $C_3H_5$   $SO_4$  +  $H_2O$ .

Ethyl sulphuric acid is an interesting compound owing to the fact that ethyl ether is prepared by converting alcohol first into this compound and then by breaking it up into ether and sulphuric acid by adding some more alcohol.

Both methyl and ethyl alcohols form sodium compounds; namely, sodium methylate, and sodium ethylate:

Ethers.—Ethers may be regarded as oxides of hydrocarbons since two like or unlike hydrocarbon radicals are connected by means of oxygen; for example, methyl ether, which is repre-

sented by the formula  $CH_3$ —O— $CH_3$ = $(CH_3)_2O$ , may be regarded as methyl oxide and analogous to sodium oxide (Na—O— $Na = Na_2O$ ).

Methyl Ether, (CH<sub>3</sub>)<sub>2</sub>O=CH<sub>3</sub>—O—CH<sub>3</sub>.—Methyl ether may be prepared by treating sodium methylate with methyl iodide:

Ethyl Ether,  $(C_2H_5)_2O=C_2H_5-O-C_2H_5$ .—Ethyl ether may be prepared by treating sodium ethylate with ethyl iodide:

In commercial practice, however, ethyl ether is made by treating alcohol with sulphuric acid. As was stated previously, when alcohol is treated with sulphuric acid, ethyl sulphuric acid

$$\begin{pmatrix} H \\ C_2H_5 \end{pmatrix}$$
 so is formed. When more alcohol is added, ethyl

sulphate is broken up into ethyl ether and sulphuric acid:

Ether thus obtained is known, in commerce, as sulphuric ether. Ether is a colorless, mobile, and fragrant liquid which boils at 34.9° C, is very inflammable, and extremely explosive when mixed with air.

Aldehydes.—The term aldehyde is derived from alcohol dehydrogenatum, which means alcohol deprived of hydrogen in consequence of oxidation. It has been demonstrated that oxygen and hydrogen in aldehyde are not connected in the form of the hydroxyl group O—H, as in alcohol, but that hydrogen is directly connected with carbon, and oxygen with carbon by its two bonds, as may be seen from the structural formula

O 
$$\parallel$$
 . The group C=O is called the carbonyl group.  $CH_s-C-H$ 

Methyl Aldehyde, HCOH.—Methyl aldehyde is known as form-

aldehyde owing to its close relation to formic acid. It is prepared by gentle oxidation of methyl alcohol. It may be obtained by passing a mixture of vapor of methyl alcohol and air over an incandescent platinum spiral, the reaction being represented by the following equation:

Methyl alcohol Oxygen Form-aldehyde Water 
$$CH_{\bullet}OH + O = HCOH + H_{2}O.$$

Methyl aldehyde is a strongly reducing agent and is extensively used as a preservative.

Ethyl Aldehyde, CH<sub>3</sub>—COH.—Ethyl aldehyde is known as acetic aldehyde, since it can be converted into acetic acid by oxidation. It is prepared by subjecting ethyl alcohol to an oxidation; for example, if we treat ethyl alcohol with a mixture of potassium dichromate and dilute sulphuric acid, acetic aldehyde is formed and can be distilled over by slightly warming on a water bath, since it boils at 21° C, and be condensed in a flask surrounded by ice-cold water. Both methyl and ethyl aldehydes are colorless and suffocating and mix with water in all proportions.

Polymerization.—When ethyl aldehyde is treated with a few drops of concentrated sulphuric acid, the liquid becomes hot. When the liquid is cooled to 0° C, the aldehyde crystallizes out. This is called paraldehyde. It melts at 10.5° C, boils at 124° C, and is very soluble in water. When the formation is accomplished at lower temperature, metaldehyde crystallizes out in needles which are insoluble in water. Both the aldehydes have the molecular formula  $(C_2H_4O)_3=C_6H_{12}O_8$ , which indicates that one molecule of either of these aldehydes is composed of three molecules of ordinary ethyl aldehyde. Such a modification is called *Polymerization*. This is a very interesting subject, owing to the fact that fructose or levulose  $(C_6H_{12}O_6)$  is artificially prepared by polymerization of methyl or formaldehyde  $(CH_2O)$ .

Acetone.—When calcium acetate (CH<sub>3</sub>COO)>Ca) is subjected to dry distillation, it breaks up into calcium carbonate and a com-

pound with the formula (CH<sub>3</sub>—CO—CH<sub>3</sub>), which is called *Acetone* or *Ketone*. Acetone may be considered as an aldehyde whose carbonyl group is united with two methyl groups instead of with one methyl group and one hydrogen atom.

Acids.—It has been demonstrated that, in organic acids, both the carbonyl group CO and the hydroxyl group OH are present

and may be represented by the structural formula 
$$\parallel$$
 C — O — H

or COOH. This resultant group, COOH, is called the carboxyl group. When a compound contains one carboxyl group, like formic acid (HCOOH), acetic acid (CH<sub>3</sub>COOH), or propionic acid (C<sub>2</sub>H<sub>5</sub>COOH), it is called a *Monobasic Acid*. When it contains two carboxyl groups, like oxalic acid ((COOH)<sub>2</sub>), malonic acid (CH<sub>2</sub>(COOH)<sub>2</sub>), or succinic acid (C<sub>2</sub>H<sub>4</sub>(COOH)<sub>2</sub>), it is called a *Dibasic Acid*. When it contains three carboxyl groups, like tri-carballylic acid (C<sub>3</sub>H<sub>5</sub>(COOH)<sub>3</sub>), it is called a *Tri-basic Acid*.

Formic Acid, CH<sub>2</sub>O<sub>2</sub>=HCOOH.—The term formic is derived from the Latin word formica (ant), in remembrance of the interesting fact that this acid was first prepared by distilling red ants. This acid can be prepared by oxidizing methyl alcohol or formaldehyde:

These reactions readily explain the relations between methyl alcohol, formaldehyde, and formic acid. In common practice, formic acid is prepared by heating oxalic acid with glycerin, the result being the decomposition of oxalic acid into formic acid and carbon dioxide:

Formic acid is a colorless liquid, has a penetrating odor, solidifies at 8.6° C, and boils at 99° C. It blisters the skin. It forms

salts with bases, such as formates of lead, copper, calcium, sodium, and potassium. They are all soluble in water.

Acetic Acid, C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>=CH<sub>3</sub>COOH.—The term acetic is derived from the Latin word acetum (vinegar), as the principal constituent of vinegar is acetic acid. Acetic acid is prepared by the oxidation of ethyl alcohol by placing in the liquid, platinum black, which acts as an oxygen carrier, converting ethyl alcohol into acetic acid:

In commercial practice, acetic acid is made by the action of air on poor wine. Air seems to carry and introduce spores of microscopic fungi known as Mother of Vinegar (Mycoderma aceti) into the alcoholic liquid where these organisms, under favorable conditions, grow, multiply, and act, in some unknown manner, as carriers of oxygen from air to alcohol. The liquid thus fermented is neutralized with soda ash, whereupon sodium acetate (CH<sub>2</sub>COONa) is formed. The solution is evaporated to dryness and then treated with sulphuric acid and distilled, when pure acetic acid passes over. The acid thus obtained is known as glacial or anhydrous acetic acid. Acetic acid is a clear, colorless liquid and has a penetrating acid odor. It solidifies at 16.7° C and boils at 119°C. It forms salts with various bases. The well known ones are acetates of sodium (CH<sub>3</sub>COONa), potassium (CH<sub>3</sub>COOK), calcium ((CH<sub>3</sub>COO)<sub>2</sub>Ca), copper ((CH<sub>3</sub>-COO), Cu), iron ((CH, COO), Fe), and lead ((CH, COO), Pb). There is a basic acetate of lead ((CH<sub>3</sub>COO), Pb-PbO) which is known as subacetate of lead and extensively used in the optical analysis of sugar for clarification of colored sugar solutions and precipitation of optically active substances other than sucrose.

When acetic acid is treated with chlorine, substitution products are formed, namely, mono-chlor-acetic acid (CH<sub>2</sub>ClCOOH) and di-chlor-acetic acid (CHCl<sub>2</sub>COOH).

Oxygen derivatives of higher hydrocarbons and benzene

series are more or less similar to those just described and are prepared in a similar manner.

Nitrogen Derivatives of Hydrocarbons.—Derivatives of hydrocarbons other than oxygen derivatives are those of nitrogen, sulphur, phosphorus, and arsenic. In this work, just amines, amido compounds, and hydrazine from nitrogen derivatives will be selected for a brief discussion, since several amido-acids are present in the composition of beets and display peculiar phenomena during the process of the purification of beet-juice and the substitution products of hydrazine are sometimes used in the analysis of sugars.

Amines.—Amines may be regarded as ammonia whose one or more atoms of hydrogen are substituted by methyl, ethyl or other hydrocarbon group, as shown by the structural formulas

Methyl-amine, NH<sub>2</sub>CH<sub>3</sub>.—When iodo-methane (CH<sub>3</sub>I) is treated with ammonia (NH<sub>3</sub>), a compound with the formula NH<sub>2</sub> CH<sub>3</sub>.HI is formed. When this compound is distilled with sodium or potassium hydroxide, methyl-amine passes over. Methylamine is a gas and liquefies at a few degrees below o°C.

Di-methyl-amine, NH(CH<sub>8</sub>)<sub>2</sub>.—Di-methyl-amine is obtained by treating di-iodo-methane with ammonia and distilling it with caustic soda or potash. It boils at 8° C.

Tri-methyl-amine, N(CH<sub>8</sub>)<sub>3</sub>.—Tri-methyl-amine is obtained by treating tri-iodo-methane with ammonia in a manner similar to the preparation of the amines described above. On a large scale, tri-methyl-amine is obtained from the waste liquids known as vinasses which give off a large quantity of tri-methyl-amine when evaporated to dryness. Tri-methyl-amine occurs widely distributed in nature. It boils at 9° C.

These amines are all strongly alkaline, dissolve in water very

readily, forming hydroxides similar to that of ammonia. They form salts with acids, as, for example:

Ethyl-amines.—When bromo-ethane  $(C_2H_5Br)$  is treated with ammonia, a bromide of ethyl-amine  $(NH_2C_2H_5.HBr)$  is formed. When this compound is distilled with caustic soda or potash, ethyl-amine  $(NH_2C_2H_5)$  passes over. Di-ethyl-amine  $(NH(C_2H_5)_2)$  is obtained by treating ethyl-amine with bromo-ethane and distilling with caustic potash. Tri-ethyl-amine is obtained in a similar manner. The properties of ethyl-amines are similar to those of methyl-amines.

Amido-compounds.—Amine may also be regarded as a hydrocarbon whose one atom of hydrogen is replaced by the group

NH, as may be seen from the formulas 
$$C < H = methyl-amine H$$

or 
$$CH_2 - C < H = \text{ethyl-amine.}$$
 The group NH, is called H

the amido group. A compound containing this group is known as an amido compound; for example, ethyl-amine may be called amido-ethane  $(C_2H_5NH_2)$ . The group NH is called the imido group; a compound containing this group, an imido compound.

Amido-acids.—Amido-acids may be regarded as an acid in which one or more atoms of hydrogen belonging to radical are replaced by amido group  $NH_2$ .

Amido-formic Acid, NH<sub>2</sub>—COOH.—This acid may be regarded as formic acid (HCOOH) whose hydrogen belonging to the radical is replaced by the amido group NH<sub>2</sub>, but it has not been observed in a free condition. Its ammonia salt, namely, am-

monium carbamate (NH<sub>2</sub>COONH<sub>4</sub>), has been prepared by bringing carbon dioxide and ammonia together:

Amido-acetic acid may be regarded as an acetic acid whose one atom of hydrogen in the radical CH<sub>8</sub> is replaced by the amido group NH<sub>2</sub>. This view is sustained by the fact that amido-acetic acid is prepared by treating a halogen derivative of acetic acid with ammonia; for example, when mono-chlor-acetic acid is treated with ammonia, amido-acetic acid and ammonium chloride are formed:

Acid Amides.—There is a class of acids whose hydroxyl group OH is replaced by the amido group NH<sub>2</sub>. They are called acid amides. When ammonium acetate (CH<sub>3</sub>COONH<sub>4</sub>) is heated, it loses water, forming a compound with the formula CH<sub>3</sub>CONH<sub>2</sub>, which may be regarded as acetic acid whose hydroxyl group OH is replaced by the amido group NH<sub>2</sub>. This compound is called Acetamide.

Amido-acids and acid-amides are interesting compounds to beet-sugar makers, owing to the fact that several of the acids derived from higher hydrocarbons and belonging to amido-acid or acid-amide or to both have been observed in beet juice. Asparagine is a fair representative of this class of compounds as it represents both amido-acid and acid-amide. It is represent-

regarded as succinic acid (C<sub>2</sub>H<sub>4</sub>< $\binom{\text{COOH}}{\text{COOH}}$ )in which one hydrogen atom of the ethylene group C<sub>2</sub>H<sub>4</sub> is replaced by NH<sub>2</sub> and the hydroxyl group OH of one of the carboxyl groups COOH is

also replaced by NH, and is scientifically called *Amido-Succinamic Acid*. When boiled with an alkali, it breaks up into aspartic acid  $(C_2H_1(NH_2) < {COOH \atop COOH})$  and ammonia. This reaction may be expressed by the following equation:

Asparagine Sodium Sodium Ammonia Water 
$$C_2H_3(NH_2)$$
  $+$  2NaOH =  $C_2H_3(NH_2)$   $+$  NH3 + H2O.

This is one of the explanations offered for the development of ammonia during the purification of beet juice with lime, for, when asparagine is boiled with lime, the following reaction takes place:

Hydrazine,  $N_2H_4=H_2N-NH_2$ .—Hydrazine is closely related to ammonia but has not been observed in the free state. Several substitution products of hydrazine have been prepared, the well known ones being ethyl hydrazine  $(H_2N-NHC_2H_5)$  and phenyl-hydrazine  $(H_2N-NHC_6H_5)$ . These substitution products bear the same relation to hydrazine that amines bear to ammonia. Phenyl-hydrazine is an interesting compound, owing to its behavior toward reducing sugars, which will be described under Carbohydrates.

Carbohydrates.—There is a class of compounds in which the hydroxyl group OH and the carbonyl group CO are present in one compound in such a manner that each individual compound may be regarded as a mixture of alcohol and aldehyde or that of alcohol and acetone. They possess characteristics of both alcohol and aldehyde or those of alcohol and acetone. They are regarded as mixed compounds and are known as Carbohydrates. Those which possess characteristics of alcohol and aldehyde are called Aldoses; those which possess characteristics of alcohol and acetone, Cetoses. Sugars, starch, cellulose, and gums belong to the class of carbohydrates. The term carbohydrate originated from the fact that these compounds are composed of carbon, hydrogen,

and oxygen and the last two elements are present in the same proportion as in the composition of water, as may be seen from the following formulas:

Cane-sugar 
$$C_{11}H_{22}O_{11} = C_{12}(H_{2}O)_{11};$$
  
Glucose  $C_{6}H_{12}O_{6} = C_{6}(H_{2}O)_{6};$   
Starch, cellulose  $C_{6}H_{1}^{1}O_{5} = C_{6}(H_{2}O)_{6}.$ 

General Properties of Carbohydrates.—There are general characteristics peculiar to carbohydrates; namely, 1, their influence on polarized light; 2, their action on Fehling's solution; 3, their behavior toward phenyl-hydrazine; 4, fermentation.

Optical Activity of Carbohydrates.—Many carbohydrates are optically active, that is, they rotate the plane of polarized light. The principles of the polarization of light and the rotation of the plane of polarized light will be discussed under Polariscope. Those substances which turn the plane of polarized light to the right are called dextro-rotatory and are designated by the prefix d, as, for example, d-glucose; those rotating the plane of polarized light to the left, levo-rotatory and are designated by the prefix l, as, for example, l-glucose; those which are indifferent to polarized light, inactive and are designated by the prefix i, as, for example, i-glucose.

Theory of Optical Activity.—As to why some substances are dextro-rotatory, while others are levo-rotatory, even when they are all of the same chemical composition, a theory has been advanced by Vant Hoff and Le Bell. When a carbon atom occupies the center of gravity of a tetrahedron at whose four corners are

carbon atom is called a symmetrical carbon atom. When these four corners are occupied by four different elements or groups, like

asymmetrical carbon atom. When these four groups are all different, with reference to the central carbon atom, there are only two different ways in which these groups can be arranged around

The difference between the two arrangements is that between either one of the two and its image in a mirror. One might be called right-handed, the other, left-handed. It has been claimed that the optical modifications of many substances are due to the influence, on polarized light, of groups differently arranged around asymmetrical carbon atoms.

Lactic acid, which is represented by the formula

activity,—dextro-rotatory, levo-rotatory, and inactive. The inactive variety is produced by mixing equal quantities of dextroand levo-varieties. These phenomena can only be explained by such supposition as cited above. It has also been claimed that all optically active organic substances contain one or more asymmetrical carbon atoms.

Specific Rotation.—Specific rotatory power is that power of a substance to rotate, in circular degrees, the plane of polarized light which passes through a column, one decimeter in length, of a solution whose I c.c. contains I gram of the substance at 20°C. Observation is usually made by a homogeneous yellow light corresponding to the Fraunhofer line D. When observation is made under these conditions, specific rotation is expressed by  $[a]_D^\infty$ , in which [a] = rotation in circular degrees, 20 = temperature in Centigrade, D, the ray of light employed; for example, the specific rotation of d-glucose is expressed by  $[a]_D^\infty = +52.74^\circ$ .

Reducing Power.—As was stated under COPPER, some sugars possess a power to reduce copper from an alkaline solution of copper sulphate which is known as Fehling's solution, the resulting product being cuprous oxide (Cu<sub>2</sub>O). For this reason, they are called Reducing Sugars.

Phenyl-hydrazine Compounds.—Another important property of some sugars is their behavior toward a compound called phenyl-hydrazine (H<sub>2</sub>N—NHC<sub>6</sub>H<sub>5</sub>). When aldehyde or acetone is treated with phenyl-hydrazine, the following reaction takes place:

When treated with an excess of phenyl-hydrazine, one molecule of glucose combines with two molecules of phenyl-hydrazine, forming a compound known as glucosazone:

These hydrazine compounds are of great importance from the scientific standpoint as they are almost insoluble in water and

can be separated from solution. Many sugars have been studied by their aid.

Carbohydrates are grouped in four classes:—I, mono-saccharides; 2, di-saccharides; 3, tri-saccharides; 4, poly-saccharides.

Mono-saccharides.—Mono-saccharides are called simple sugars. There are several well known compounds belonging to this class. They are named according to the number of atoms of oxygen; for example,  $C_2H_4O_2 = \text{diose}$ ;  $C_3H_6O_3 = \text{triose}$ ;  $C_4H_8O_4 = \text{tetrose}$ ;  $C_5H_{10}O_5 = \text{pentose}$ ;  $C_6H_{12}O_6 = \text{hexose}$  and so forth.

Diose, C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>.—Diose is obtained by oxidizing glycol with nitric acid, in which the following reaction takes place:

Diose is a very interesting compound on account of its being the simplest carbohydrate. Its molecular structure may be repre-

tion and combines with phenyl-hydrazine forming osazone.

Triose, C,H6O3.—Triose is represented by glycerose which is

said to be a mixture of glyceric aldehyde 
$$\begin{pmatrix} COH \\ CHOH \\ CH_2OH \end{pmatrix}$$
 and di-

oxyacetone 
$$\begin{pmatrix} CH_2OH \\ CO \\ CH_2OH \end{pmatrix}$$
. It is prepared by oxidizing glycerine,

which is tri-acid alcohol, represented by the formula, CHOH,

using a mild oxidizing agent such as bromine and sodium car-

bonate. It reduces Fehling's solution, forms osazone, and is susceptible to fermentation.

Tetrose, C<sub>4</sub>H<sub>8</sub>O<sub>4</sub>.—A representative of this sugar is erythrose,

rose is obtained by mild oxidation of erythrite which is tetra-acid

Pentose, C<sub>5</sub>H<sub>10</sub>O<sub>5</sub>.—There are two well known sugars belonging to this group,—arabinose and xylose. Arabinose, which is

pared by boiling cherry gum with dilute sulphuric acid. Arabinose can be converted into arabite, which is pent-acid alcohol and

reduction. Arabinose reduces Fehling's solution, forms phenylhydrazone, is fermentable, and is optically active, its specific rotation being  $[a]_D^\infty = + 105.4^\circ$ . It is, however, called *l*-arabmose owing to its relation to levulose and levo-mannose. *Xylose*,

is obtained by boiling wood gum with any dilute acid. It can

be converted into xylite, which is pent-acid alcohol and repre-

tion. Xylose reduces Fehling's solution, forms phenyl-hydrazone, and is optically active, its specific rotation being  $[a]_D^\infty = +19.51^\circ$ .

Hexose, C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>.—There are five well known sugars belonging to this group,—glucose, fructose, mannose, galactose, and invert sugar.

Glucose is known by several different names:—glucose, glycose, grape-sugar, or starch-sugar. Glucose occurs in nature very widely distributed together with fructose. It is found in sweet fruits, honey and also in blood, liver, and urine. It is obtained by treating starch or cellulose with dilute sulphuric acid. The acid seems to carry molecules of water into the molecular structure of starch in order to form glucose. This process is called Hydrolysis. The reaction may be represented by the following equation:

It has been demonstrated that glucose is an aldose and is repre-

It is interesting to note that, when oxidized by bromine or chlorine, glucose is converted into gluconic acid

and when oxidized by nitric acid, it is transformed into saccharic acid

$$\begin{bmatrix}
H & H & OH & H \\
 & | & | & | & | \\
 COOH - C - C - C - C - C - COOH \\
 & | & | & | & | \\
 OH & OH & H & OH
\end{bmatrix},$$

and that, by reducing with nascent hydrogen, glucose is converted into hex-acid alcohol, sorbite

There are several compounds of glucose with metals and salts. The best known ones are:

Sodium glucose  $C_6H_{11}O_6Na$ , Sodium chloride glucose  $C_6H_{12}O_6\cdot NaCl+1/2 H_2O$ , Sodium chloride glucose  $C_6H_{12}O_6\cdot 2NaCl$ , Cupric oxide glucose  $C_6H_{12}O_6\cdot 5CuO$ .

Glucose crystallizes out from concentrated solution in masses of minute six-sided plates. A solution of glucose suffers fermentation, producing alcohol and carbon dioxide. It reduces Fehling's solution and forms phenyl-hydrazone and osazone. It is optically active, its specific rotation being  $[a]_D^\infty = +52.74^\circ$  when polarized in 10 per cent solution. The sweetness of glucose is about 6/10 that of cane-sugar.

In commercial practice, glucose is made from corn or potato starch by heating with dilute sulphuric acid, which is removed after hydrolysis is completed, by mixing the solution with chalk and then filtering off. The solution thus obtained is evaporated to a syrup and sent to market as glucose, or, to dryness and sold as grape-sugar.

There are two other varieties belonging to this group, l-glucose and i-glucose. The specific rotation of l-glucose is  $[a]_D^\infty = -51.4^\circ$  when polarized in 4 per cent solution; i-glucose is inactive.

Fructose is also known by other names:—levulose and fruitsugar. As was stated under glucose, fructose occurs in fruits together with glucose. It is prepared by oxidizing mannite, which is hex-acid alcohol with the formula,

It has been demonstrated that fructose is a cetose and is represented by the formula,

$$CH,OH - C - C - C - CO - CH,OH.$$

When reduced, fructose is transformed into mannite. It is also prepared by treating inulin  $(C_0H_{10}O_5)$  with dilute acid. Fructose reduces Fehling's solution, forms phenyl-hydrazone, is fermentable and optically active, its specific rotation being  $[a]^{\infty} = -92.0^{\circ}$  to  $-92.5^{\circ}$  when polarized in 10 per cent solution. Ordinary fructose, although it is levo-rotatory, is called d-fructose on account of its being related to dextro-rotatory substances. Its sweetness is about the same as that of cane-sugar.

There are two other varieties belonging to this group, namely, l-fructose and i-fructose. l-Fructose is dextro-rotatory and not fermentable; i-fructose, inactive and partially fermentable. It is extremely interesting from the scientific standpoint to note that i-fructose is obtained by the polymerization of formaldehyde and also by treating glycerose with a weak aikali:

Formaldehyde 6CH <sub>2</sub> O	=	i-fructose C <sub>6</sub> H <sub>12</sub> O <sub>6</sub> ;
Glycerose		z-fructose
2C <sub>8</sub> H <sub>6</sub> O <sub>3</sub>	=	$C_6H_{12}O_6$ .

Invert Sugar is prepared by hydrolyzing cane-sugar in which the following reaction takes place:

As may be seen from this equation, one molecule of cane-sugar, on acquiring one molecule of water, has been split into two molecules of sugar of the formula  $C_6H_{12}O_6$ . It has been demonstrated that one of these two is glucose or dextrose and the other, fructose or levulose. Levulose forms a compound with lime insoluble in water. This fact is taken advantage of in separating levulose from dextrose. Ten parts of invert sugar are dissolved in 100 parts of water and the solution is cooled with ice and 6 parts of powdered lime added, whereupon levulose is precipitated while glucose remains in solution. The precipitate is filtered off and thoroughly washed and treated with carbon dioxide, which sets levulose free.

The specific rotation of invert sugar is  $[a]_D^{\infty} = -19.447^{\circ}$ . The levo-rotatory power of invert sugar is due to the higher specific rotatory power of levulose than that of dextrose. Other properties of invert sugar are the same as those of glucose and fructose.

Mannose.—d-Mannose, which is represented by the formula,

mannite or hydrolyzing cellulose. Its specific rotation is  $[a]_D^\infty = +$  12.96° when polarized in 10 per cent solution. It reduces Fehling's solution, forms osazone, and is fermentable. *l-Mannose* and *i-mannose* have also been prepared.

Galactose.—d-Galatose is obtained by hydrolyzing milk-sugar and is represented by the formula,

Its specific rotation is  $[a]_D^{20} = +81.2^{\circ}$ . It reduces Fehling's solution, forms osazone, and is not fermentable.

Di-saccharides.—Sugars of this class have the general formula  $C_{12}H_{22}O_{11}$  and may be regarded as those built up from two

molecules of simple sugar, hexose with the formula  $C_8H_{12}O_6$ , after being derived of one molecule of water:

It is for this reason that they are called di-saccharides. The most commonly known sugars of this class are cane-sugar, milk-sugar, and maltose.

Cane-sugar.—Cane-sugar will be discussed in a special chapter.

Milk-sugar.-Milk-sugar is called, in chemistry, lactose and is represented by the formula C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>. Milk of all mammals contains lactose. In the manufacture of cheese, casein is precipitated by means of rennet and filtered off. The filtrate contains milk-sugar. When evaporated down, the sugar crystallizes out and may be purified by recrystallization. Lactose crystallizes in rhombic crystals with one molecule of water (C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>.H<sub>2</sub>O). When heated to 145° C, lactose loses water of crystallization and begins to decompose. It is optically active, its specific rotation being  $[a]_D^{20} = +52.53^{\circ}$  (Schmöger). It reduces Fehling's solution, forms osazone. It ferments with the formation of lactic acid, which causes coagulation of the casein in the milk. When oxidized with nitric acid, it is converted into mucic and saccharic acids. On being reduced with nascent hydrogen, it is converted into mannite (C<sub>6</sub>H<sub>8</sub>(OH)<sub>6</sub>) and dulcite (C<sub>6</sub>H<sub>8</sub>  $(OH)_a$ ).

Maltose.—When barley is steeped in water until it germinates and then dried, the product is called malt. Malt contains a substance known as diastase whose behavior toward starchy matter is similar to that of ferments. When treated with malt, starch is transformed into a sugar with the formula  $C_{12}H_{22}O_{11}$  and dextrin  $(C_6H_{10}O_5)$ . The sugar thus obtained is called Maltose. The reaction may be expressed by the following equation:

Starch  $_{3}C_{6}H_{10}O_{5}$  +  $_{12}W_{12}O_{12}$  =  $_{12}M_{12}O_{11}$  +  $_{12}O_{12}O_{13}$  +  $_{14}O_{5}$ . When oxidized with nitric acid, maltose is converted into d-saccharic acid. When hydrolyzed with sulphuric acid, it is converted into d-glucose. Maltose ferments with yeast, being converted first into glucose and then into alcohol. It crystallizes in fine

needles with one molecule of water of crystallization  $(C_{12}H_{22}O_{11}.H_2O)$  which it loses when heated at 100°C. It reduces Fehling's solution and forms osazone. It is dextro-rotatory, its specific rotation being  $[a]_D^{*o} = + 138.29^{\circ}$  when polarized in 10 per cent solution of water (Herzfeld).

Tri-saccharides.—Sugars of this class are represented by the general formula  $C_{18}H_{82}O_{16}$  and may be regarded as built up from three molecules of simple sugar, hexose  $(C_6H_{12}O_6)$ , after being deprived of two molecules of water, thus:

The best known sugar of this class is raffinose.

Raffinose.—Raffinose is also called melitose, melitriose, or gossy-The term raffinose is derived from the French word raffiner (to refine), owing to the fact that it was identified first in the products of beet-sugar refining. Raffinose is found in sugar-beets in small quantities and also in cotton seeds. Beets contain as high as 0.02 per cent raffinose. It is much more readily soluble in hot water than sucrose, consequently it remains in syrups and molasses during sugar refining process, especially in those products obtained from the process of extracting sugar from molasses. It is claimed that, when present in large quantity, it modifies the crystal forms of sucrose. gives the influence of raffinose on the crystallization of cane-sugar at various percentages. When I per cent of raffinose to canesugar is present in a solution, cane-sugar crystallizes out in usual forms; with 3 per cent of raffinose, cane-sugar crystals become somewhat elongated; with from 5 to 7 per cent, they assume needle shape; with higher percentage, up to 12 per cent, they become fine and sharp-pointed; with 25 per cent, crystallization ceases. Moreover raffinose is strongly optically active and causes difficulties in sugar analysis. For these reasons, it is very important for the sugar-house chemist to be thoroughly familiar with the properties of raffinose.

Raffinose is represented by the formula  $C_{18}H_{82}O_{16}$ , but it crystallizes out from water solution with 5 molecules of water of crystallization, as represented by the formula  $C_{18}H_{82}O_{16}$ ,  $5H_2O$ ,

which is called hydrate. On being heated slowly between 100° and 105° C, it loses water of crystallization and is called anhydride. On being heated to 130° C, it decomposes. In cold water, it is difficultly soluble, but in hot water, more easily soluble than cane-sugar. It dissolves easily in absolute methyl alcohol. This fact is taken advantage of in separating raffinose from canesugar as the latter is insoluble in methyl alcohol. Raffinose is insoluble in absolute ethyl alcohol.

In water solution, lead acetate has no effect on raffinose. In the presence of ammonia and alcohol or methyl alcohol, however, lead acetate precipitates a compound with the formula  $C_{18}H_{82}O_{16}$ . 3PbO which is insoluble in water and alcohol. There are two lime compounds of raffinose with the formulas  $C_{18}H_{32}O_{16}$ .2CaO.  $5H_2O$  and  $C_{18}H_{32}O_{16}$ .3CaO.3 $H_2O$ ; the former is soluble and the latter insoluble in water. When boiled with caustic alkali or lime, raffinose remains unaffected. When oxidized with strong nitric acid, it is converted into oxalic, saccharic, and mucic acids. By inversion or hydrolysis, it is converted into three simple sugars,—d-fructose, d-glucose, and d-galactose:

Raffinose Water d-Fructose d-Glucose d-Galactose 
$$C_{18}H_{32}O_{16}$$
 +  $2H_2O$  =  $C_4H_{12}O_6$  +  $C_6H_{12}O_6$  +  $C_6H_{12}O_6$ .

Raffinose is strongly dextro-rotatory, its specific rotation being  $[a]_D^{20} = + 104.5^{\circ}$  for the hydrate  $(C_{18}H_{32}O_{16}.5H_2O)$  when observed in 10 per cent solution. 26.048 grams of raffinose hydrate, dissolved in 100 c. c. of pure water at 17.5° C, polarized at 20° C in 200 m. m. observation tube, rotate the plane of polarized light, to the right, 157.15° on the Venzke scale. anhydride, under the same conditions, rotates Hence the rotatory power of raffinose hydrate is 1.57 times and that of raffinose anhydride 1.87 times that of cane-sugar, since cane-sugar, under the same conditions, rotates exactly 100°. These relative optical activities are of great value they are used as factors in the calculation of sugar and raffinose in the inversion method, which will be described in the chapter on Special Analysis.

Raffinose does not reduce Fehling's solution, but is readily fermentable.

Poly-Saccharides.—Compounds belonging to this class are represented by the general formula  $(C_6H_{10}O_5)_n$  and may be regarded as those built up from n molecules of simple sugar, hexose  $(C_6H_{12}O_6)$  after being deprived of n molecules of water:

Hexose Water Poly-saccharide 
$$nC_6H_{12}O_6$$
 —  $nH_2O$  =  $(C_6H_{10}O_5)_n$ .

The molecular formulas of these compounds have not yet been established, although the analytical results indicate that carbon, hydrogen, and oxygen are combined in such a proportion as shown by the formula  $C_6H_{10}O_5$ . It is highly probable that the molecular formulas of these compounds are multiples of  $C_6H_{10}O_5$ , as, for example,  $(C_6H_{10}O_5)_4$ ,  $(C_6H_{10}O_5)_5$ , or  $(C_6H_{10}O_5)_6$ . The best known compounds of this class are cellulose, starch, dextrin, and gums.

Cellulose, (C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>).—All vegetable tissues consist of minute cells, the walls of which are essentially cellulose. According to various sources, they differ in appearance and physical properties, but the chemical composition and properties are always the same. The difference in appearance is due to the presence of other substances, such as oil, coloring matter, starch, and gum. They may be removed by chemical treatment. Cotton and hemp consist nearly entirely of cellulose. Pure cellulose may be prepared by treating cotton successively with ether, alcohol, water, caustic alkali, and dilute hydrochloric acid and then washing with water.

Cellulose is insoluble in water, alcohol, or ether. It dissolves in a liquid known as Schweitzer solution, which is made by dissolving pure copper hydroxide in ammonia. It also dissolves in concentrated sulphuric acid. When the sulphuric acid solution of cellulose is diluted and boiled, cellulose will be converted into dextrin and glucose:

Cellulose Water Glucose Dextrin 2 
$$C_6H_{10}O_5$$
 +  $H_2O$  =  $C_6H_{12}O_6$  +  $C_6H_{10}O_5$ .

It may be seen from this reaction that glucose may be manufactured from wood, straw, paper, rags, or any other material consisting mainly of cellulose.

Gun-cotton.—When treated with a mixture of nitric and sulphuric acids, cellulose forms nitro-cellulose ( $C_{12}H_{14}O_4(ONO_2)_4$ ) which is known, in commerce, as gun-cotton and is extensively used as an explosive.

Paper.—The essential constituent of paper is cellulose; it is usually made from cotton and linen rags, wood, or straw. These materials are distintegrated partly by mechanical means, partly by boiling with caustic soda and are finally reduced to pulp by means of knives. The pulp thus obtained is passed through between rollers with a required amount of water in order to produce paper.

Starch,  $(C_0H_{10}O_5)_n$ .—Starch occurs very widely distributed in nature, having been found in all plants in greater or less quantities. It is found in large quantities in wheat, corn and potatoes, the last two being the chief sources from which starch is manufactured. The corn is soaked in warm water and ground in a stone-mill with a constant stream of water in order to carry away the starch. The milky liquid, running from the mill, is filtered through a silk sieve and allowed to stand so that the starch may settle down and the water be drawn off. The starch thus obtained is treated with a very dilute solution of caustic soda in order that it may be freed from foreign substances, such as glutin and oil, and washed thoroughly with water and dried at low temperature.

Starch, when examined under the microscope, consists of minute granules called starch-grains, which are insoluble in cold water. When boiled in water, the walls of starch-grains break up and the contents partially dissolve in water. When cooled, the solution becomes a colorless gelatinous fluid called starch paste. Iodine gives a blue color to a starch solution; bromine, a yellow color. Starch does not reduce Fehling's solution.

As stated previously, starch, when treated with a dilute acid, is converted into glucose. When treated with a diastase, however, starch is converted into maltose and dextrin. This transformation may be proved by testing starch paste with Fehling's solution before and after the treatment thereof with the

reagents just cited. The specific rotation of potato starch is given as  $[a]_D = +202^{\circ}$  (Brown, Morris, and Miller).

Dextrin,  $(C_0H_{10}O_5)_n$ .—Dextrin may be obtained by heating starch at between 170° and 200° C. It may also be obtained, as stated previously, by treating starch with a diastase. When boiled with an acid for a long time, dextrin is converted into glucose. It is an amorphous, gummy matter, soluble in water but insoluble in alcohol. It does not crystallize. It is extensively used as a substitute for gum, being commonly known as starch-gum or British gum. It is called dextrin on account of its dextro-rotatory action on polarized light, its specific rotation being  $[a]_1 = +206.5^\circ$  (Brown and Morris). It gives a red color with iodine. The molecular formula is claimed by Brown and Morris to be  $C_{12}H_{22}O_{11}.6C_{12}H_{20}O_{10}$ .

Inulin,  $(C_6H_{10}O_5)_n$ .—Inulin is very widely distributed in nature, being found in the roots of elecampane, dandelion, and chicory, in the tubers of potatoes, in the seeds of sun-flower and many other plants. It may be obtained by boiling the roots of elecampane with water in the presence of sodium carbonate. The solution thus obtained is cooled with a freezing mixture, whereupon inulin is precipitated. It is purified by redissolving in hot water and precipitating it by cooling with a freezing mixture. Inulin is a soft, tasteless, amorphous powder, insoluble in alcohol, slightly soluble in cold water, but very readily soluble in boiling water. It gives a yellow color with iodine. It is levo-rotatory, its specific rotation being  $\begin{bmatrix} a \end{bmatrix}_{ij}^n = -36.66^\circ$  (Kiliani). When treated with a dilute acid, it is converted into levulose. Its molecular formula is claimed by Kiliani to be  $C_{86}H_{62}O_{81} = 6C_6H_{10}O_6.H_2O$ .

Gums.—There are several varieties of gums, the best known ones being gum arabic and wood-gum. The general formula of gums is  $C_{12}H_{22}O_{11}$ , but Landolt assigns  $C_{10}H_8O_9$  to gum arabic and  $C_8H_8O_4$  to wood-gum.

Gum Arabic.—Gum arabic is also known as arabin or arabic acid. It is obtained from Acacia arabica (gum arabic tree) which grows abundantly in India and Arabia. It is brittle, in-

odorous, and has a mucilaginous taste. It is insoluble in alcohol, dissolves in water but precipitates when hydrochloric acid is added. When arabin is hydrolyzed, pectine-sugar or arabinose is formed. When boiled with strong nitric acid, it is converted into mucic, saccharic, and oxalic acids. Arabin can be distinguished from dextrin by the reaction that the former gives no dextrose on hydrolization.

It has been found by Scheibler that the gums called meta-pectic acid occurring in sugar-beets are identical with arabin or gum arabic. Arabin occurring in beets has been found to be in an insoluble form, becoming only steeped on coming in contact with water. Gums contained in spoiled beets or even those found in sound beets of certain years are readily soluble in water and go directly into the diffusion juice. It is also likely that some portion of the insoluble gums may become finely divided in consequence of remaining in water for a long time and be carried into the juice. The juice containing arabin in a large quantity can readily be recognized by its foaming, turbid appearance and slow and difficult boiling.

Arabin of the insoluble variety is rendered soluble by the action of alkalies. Milk of lime does not precipitate arabin but basic calcium salt of arabin is insoluble in water. Lead acetate does not precipitate arabin except in the presence of alcohol or ammonia. Arabin rotates the plane of polarized light to the left, its specific rotation being  $[a]_D = -98.5^{\circ}$ .

Wood-gum.—Wood-gum is called, in chemistry, xylon which is derived from the Greek word xulon (wood). It is obtained from birch, ash, beech, and many other trees. As stated previously, wood-gum changes into xylose when hydrolyzed. It is levo-rotatory, its specific rotation being  $\lceil \alpha \rceil_D = -84.0^\circ$ .

## Chapter V.

## CANE-SUGAR.

This chapter will be devoted to the discussion of the properties of cane-sugar, Nature's mode of preparation thereof, the composition of beets, the purity of beet-juice, and the constituents of non-sugars occurring in beet-juice.

Occurrence.—Cane-sugar occurs very widely distributed in nature. It is found, in abundance, in the cell-sap of sugar-cane, (Saccharum officinarum), sugar-beet (Beta cycla or Beta vulgaris), Indian corn, sugar-maple, sorghum (Sorghum saccharatum), and date-palm. It is also found in nearly all higher plants in greater or less quantities.

History.—The cultivation of sugar-cane had been known to the Chinese and the Hindoos in remote ages. The extraction of sugar from sugar-cane was first practiced in Arabia and Egypt. The industry of cane-sugar manufacture was introduced into Sicily in 1230 and soon afterwards to the Canary Islands. The introduction of this industry to the West Indies was accomplished during the 16th century when it became a very important industry commercially.

The manufacture of sugar from beets dates back to the middle of the 18th century. A. S. Marggraf, Director of the Physical Class of the Academy of Science at Berlin, discovered the presence of cane-sugar in red beets in 1747 and recommended the cultivation thereof. Franz Carl Achard, Director of the Physical Class of the Academy of Science at Berlin, succeeded in establishing the first beet-sugar factory in 1799 at Steinau, Germany Since then, the beet-sugar industry made great progress. To-day Europe alone produces annually nearly 7 million tons of beet-sugar.

Formula and Name.—The formula of cane-sugar is represented by  $C_{12}H_{22}O_{11}$ . Several structural formulas have been

proposed for cane-sugar. The following is one of the formulas worked out by Zincke:

Cane-sugar is called, in chemistry, sucrose or saccharose. It is also known as beet-sugar. The term cane-sugar was originated from the historical fact that this sugar was first prepared from cane. When the term sugar without a prefix appears in this work, it should be understood to have reference to this particular sugar.

Physical Properties.—Cane-sugar crystallizes in the mono-

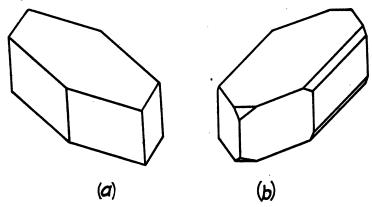


Fig. 2.—Crystals of Cane-sugar.

clinic system. (a) in Fig. 2 shows the fundamental form and (b), the combination form of usual occurrence. The crystals are formed as bright, short, and oblique prisms which are truncated on their edges and corners in various shapes. They are perfectly transparent when in a pure state. The presence of other substances modifies the crystal forms of cane-sugar; for example, raffinose imparts needle shape to cane-sugar crystals under certain circumstances. Crystals of cane-sugar contain no water of crystallization, and resist the action of the atmosphere. They have a peculiar property of producing electric light when broken in the dark. The specific gravity of pure sucrose is 1.61.

Sucrose has a sweet taste. It is readily soluble in water; the solubility increases with the temperature; at 10°C, water holds 66 per cent sugar in solution; at 40°C, 76 per cent; at 50°C, 83 per cent. Sucrose is insoluble in absolute alcohol and absolute methyl alcohol. In dilute alcohol, the solubility increases with the amount of water present. Sucrose may be crystallized out from a saturated solution by cooling or by further evaporating in order to concentrate the solution to supersaturation. The purer the solution, the easier the formation of crystals. The presence of invert sugar, salts, and organic non-sugars hinders the crystallization of sucrose.

Crystals of sucrose have no effect on polarized light. Its solution, however, rotates the plane of polarized light to the right, its specific rotation being  $[a]_{\mathcal{D}}^{\infty} = +$  66.5.° This is a very important property, owing to its application to the quantitative estimation of sucrose and will be discussed further under the Polariscope, somewhat in detail. The presence of alcohol in the solvent has no effect on the optical activity of sucrose. The presence of carbonates, acetates, sulphates, and chlorides of alkaline metals and also free alkalies, lime, baryta and strontia diminishes the optical activity of sucrose to greater or less degree. In a water solution, basic acetate or subacetate of lead does not affect the rotatory power of sucrose. In a strong alcoholic solution, however, its rotatory power is diminished by lead salts, owing to the formation of insoluble lead saccharate.

Chemical Properties.—When dried between 100° and 110°C, sucrose remains unchanged; when the temperature is gradually increased, it begins to melt at 160°C, undecomposed, to a clear liquid which solidifies on cooling to an amorphous glassy mass and will, after a short time, assume crystalline structure and consequently become opaque. Through further increase of temperature, decomposition takes place between 200° and 210°C, when a considerable quantity of gas is given off and a darkbrown colored and bitter tasting product is formed. This product is called caramel or caramelized sugar. At a still higher temperature, the mass will swell, spatter and blacken,

evolving a large quantity of a partly combustible gas with a disagreeable odor and finally leaving behind a bright, extremely hard, and difficultly combustible, carbonaceous matter.

When a neutral water solution of sucrose is heated at 70°C for a short time, no appreciable decomposition takes place. When it is boiled at a higher temperature for a longer duration, a gradual decomposition takes place.

·Strong oxidizing agents decompose sucrose completely; when warmed with moderately concentrated nitric acid, sucrose is converted into saccharic acid  $\left(C_4H_4(OH)_4 < {COOH \atop COOH}\right)$ . When

boiled with concentrated nitric acid, sucrose changes into oxalic acid with an evolution of red fumes. Concentrated sulphuric acid chars sugar with development of a very high temperature and a vigorous evolution of sulphur dioxide, formic acid, carbon dioxide, and carbon monoxide. Concentrated hydrochloric and phosphoric acids also decompose sugar.

Dilute mineral acids, namely, sulphuric, hydrochloric, hydrofluoric, nitric, and phosphoric acids and also strong organic acids, such as oxalic, salicylic, citric, and tartaric acids decompose sugar in such a manner that, on acquiring one molecule of water, it splits itself into glucose and fructose. This change is called *Inversion*. Inversion takes place at ordinary temperature slowly, but can be effected in a shorter time by boiling. Carbon dioxide and sulphur dioxide, under ordinary circumstances, invert sucrose in water solution in a small degree, but completely under pressure and high temperature. When a sugar solution is boiled with a weak acid for a long time, sugar is decomposed completely with the formation of a brown humus substance.

When a sugar solution, mixed with  $\alpha$ -naphthol and alcohol, is brought in contact with pure concentrated sulphuric acid, a characteristic color is produced varying, from dark red to clear rose, according to the amount of sugar present. This reaction, being very delicate and sensitive, is used for detecting a very small quantity of sugar.

Sucrose does not ferment until it is converted into invert

sugar by the action of the yeast plant or invertin separated from yeast or by means of some acids. The fermentation products of sugar are not as simple as usually supposed, many substances besides alcohol and carbon dioxide being formed. According to Pasteur, 100 parts of sucrose, which correspond to 105.26 parts of invert sugar, give 51.11 parts alcohol, 49.42 parts carbon dioxide, 0.67 part succinic acid, 3.16 parts glycerine, and 1 part the matter consumed by the yeast.

Compounds.—Sucrose combines with the oxides of potassium, sodium, calcium, barium, strontium, lead, and certain other metals, forming salt-like compounds in which sucrose plays the rôle of a weak acid. These compounds are called saccharates or sucrates. According to their composition, there are several kinds of saccharates, namely, mono-basic, di-basic, and tri-basic saccharates. In general, saccharates are unstable, not of sweet taste, partly readily soluble, and partly difficultly soluble in water. They are all soluble in dilute acids whereby sucrose is set free.

Baryta Saccharate.—With baryta (BaO), sucrose forms a compound with the formula  $C_{12}H_{22}O_{11}BaO$ , which may be obtained in crystalline precipitates by mixing a solution of baryta with that of sucrose and heating. In cold water, baryta sucrate remains in solution but can be precipitated by adding alcohol. Baryta sucrate may be decomposed by treating with carbon dioxide. This principle is applied in a process of obtaining sugar from low grade syrups. Baryta sucrate dissolves in 41 times its own weight of water at 15°C and in 43 times at 100°C.

Strontia Saccharate.—When a solution of strontium hydroxide is added to a boiling solution of sugar in such a proportion that two molecules of strontia (SrO) may combine with one molecule of sucrose, we obtain di-basic strontium sucrate with the formula  $C_{12}H_{22}O_{11}2SrO$ . When cooled, this compound is decomposed, the product being mono-basic strontium sucrate ( $C_{12}H_{22}O_{11}SrO$ ) which decomposes at 58°C. According to Scheibler, one liter of water at 10°C holds, in solution, 37.5 grams of mono-basic strontia sucrate; at 20°C, 48.6 grams; at

30°C, 62.7 grams; at 40°C, 82.6 grams; at 50°C, 121.9 grams; at 58°C, 185.1 grams.

Calcium Saccharates.—Lime forms with sucrose four well known sucrates:—Mono-calcium saccharate, di-calcium saccharate, sesqui-calcium saccharate, and tri-calcium saccharate. These sucrates are of special interest to sugar experts, as the Steffen process of recovering sugar from low grade syrups is based on the formation of saccharates of lime.

Mono-calcium Saccharate, C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>CaO.—Mono-calcium saccharate is obtained by mixing a sugar solution with milk of lime. After filtration, it is precipitated by adding alcohol to the filtrate. It is soluble in cold water. When boiled, it is converted into tri-calcium saccharate and sucrose:

Mono-calcium saccharate Sucrose 
$$3C_{12}H_{12}O_{11}CaO = C_{12}H_{22}O_{11}3CaO + 2C_{12}H_{22}O_{11}$$

Mono-calcium saccharate crystallizes out from cold water with two molecules of water:— $C_{12}H_{22}O_{11}CaO+2H_2O$ .

Di-calcium Saccharate, C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>2CaO.—Di-calcium saccharate is obtained by treating a sugar solution with an excess of lime and precipitating with alcohol. It separates in white crystals which are soluble in 33 times its own weight of water and are very readily soluble in sugar solution. When a solution of di-calcium saccharate is boiled, it is converted into tri-calcium saccharate, soluble di-calcium saccharate, and sucrose.

Sesqui-calcium Saccharate, 2C<sub>12</sub>H<sub>22</sub>O<sub>113</sub>CaO.—This saccharate is obtained by adding an excess of milk of lime to a strong solution of sugar and evaporating it to dryness.

Tri-calcium Saccharate, C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>3CaO.—Tri-calcium saccharate is formed by boiling di-calcium saccharate:

Di-calcium saccharate 
$$^{\text{Tri-calcium}}$$
 saccharate  $^{\text{Mono-calcium}}$  saccharate  $^{\text{2C}}_{12}H_{12}O_{11}^{2}CaO$  =  $C_{12}H_{12}O_{11}^{2}CaO$  +  $C_{12}H_{12}O_{11}^{2}CaO$ .

Tri-calcium saccharate dissolves in 100 parts of cold and 200 parts of boiling water and is readily soluble in a sugar solution and slightly soluble in alcohol. On standing in water, it decom-

poses with the formation of calcium hydroxide and mono-calcium saccharate.

Tetra-calcium, hexa-calcium, octa-calcium saccharates, and a compound with the formula  $C_{12}H_{20}O_{11}Ca$  have also been observed.

All these calcium saccharates are decomposed by acids. Carbon dioxide and sulphur dioxide also decompose these saccharates. The process of saturation is based on this property of saccharates. When carbon dioxide gas is passed into a thick solution of sucrate of lime, there will first be formed a gelatinous mass, which is called by Boivin and Loiseau, Hydrosucrocarbonate and is represented by the formula  $3CO_3CaO+C_{12}H_{22}O_{11}3CaO+2H_2O$ . Horsin-Deon has obtained a sucrocarbonate of lime with the formula  $3CO_3CaO+C_{12}H_{22}O_{11}CaO+H_2O$ . These sucrocarbonates are decomposed by an excess of carbon dioxide or sugar or by the influence of temperature higher than that which existed at the time of their formation.

The rotatory power of sucrates is lower than that of sucrose whose quantity corresponds to that contained in sucrate. For this reason, in the polarimetric analysis of sucrose, a solution containing sucrate should be neutralized with an acid before polarization.

Solubility of Oxides of Other Metals in Sugar Solution.— Oxides of certain other metals are soluble in the solution of sucrate of lime in the presence of an excess of sugar. Among the most common are oxides of magnesium, iron, aluminum, manganese, zinc, copper, and lead. Hydroxides of iron and aluminum are also soluble in sugar solution. Metallic iron dissolves gradually in a sugar solution when allowed to have a free access to the air and turns the color of the solution red-brown. When the solution is evaporated, an amorphous residue of sucrate of iron oxide is left behind. These oxides and hydroxides could not be precipitated from a sugar solution by means of hydroxides of ammonium, sodium, or potassium. Whether there exists sucrate of magnesia or not, is not known. Magnesia, however, is slightly soluble in a sugar solution and is readily soluble in a

solution of sucrate of lime. Metallic copper also dissolves in sugar solution in the presence of air. Copper carbonate too behaves in a similar manner. It is probable that the solubility of these oxides in a sugar solution is due to the formation of saccharates of these oxides similar to those of lime.

Sucrate of Copper Sulphate (CuSO<sub>4</sub>+C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>+4H<sub>2</sub>O). —This compound is obtained by saturating a solution with copper sulphate and sucrose.

Saccharates of Lead.—There are two well known sucrates of lead:—di-plumbic sucrate and tri-plumbic sucrate. Di-plumbic sucrate ( $C_{12}H_{18}Pb_2O_{11}$ ) may be prepared by boiling litharge (PbO) in a sugar solution. It may also be obtained by treating an ammoniacal solution of neutral lead acetate with a sugar solution. Tri-plumbic sucrate ( $C_{12}H_{18}Pb_3O_{11}$ ) may be prepared by adding a certain quantity of caustic potash or soda to a mixture of sugar and neutral lead acetate solution and heating. It may also be obtained by adding ammoniacal solution of lead acetate to a sugar solution until a permanent precipitate is formed. Tri-plumbic sucrate is readily soluble in a sugar solution, insoluble in cold, but slightly soluble in hot water.

Saccharates of Sodium and Potassium.—When an alcoholic solution of sugar is treated with caustic soda, sodium sucrate with the formula  $C_{12}H_{21}NaO_{11}$  is formed, and with caustic potash, potassium sucrate with the formula  $C_{12}H_{21}KO_{11}$ .

Saccharates of Sodium and Potassium Compounds.—Sodium chloride forms, with sucrose, compounds with the following formulas:  $C_{12}H_{22}O_{11}NaCl$ ,  $C_{12}H_{22}O_{11}NaCl + 2H_2O$ , and  $2C_{12}H_{22}O_{11}+3NaCl+4H_2O$ ; potassium chloride forms a compound with the formula  $C_{12}H_{22}O_{11}KCl$ ; sodium iodide,  $2C_{12}H_{22}O_{11}3NaI + 3H_2O$ ; sodium bromide,  $C_{12}H_{22}O_{11}NaBr+1 1/2H_2O$ .

Nature's Mode of Manufacturing Sugar.—It would be interesting to consider briefly the mode in which Nature manufactures sugar as it may help sugar-beet planters in growing and taking care of beets. This will be discussed under two headings:—plant-cells and growth of beet.

Plant-cells.—An examination of a thin section of any soft and growing part of some plant under a powerful microscope reveals that it consists of small sacs which vary considerably in shape and size according to the sources of the materials. These sacs are called Plant-cells. A careful observation shows that all these sacs contain a slimy, semifluid substance which is called protoplasm. Protoplasm is not homogeneous throughout. It consists, as shown in Fig. 3, of cell-wall, cytoplasm, nucleus, nucleole, chromatophores, and centrospheres. The cell-wall is composed essentially of cellulose. The exact chemical composition of protoplasm is not known. It appears, however, highly probable that it is a mixture of several compounds composed of carbon, hydro-

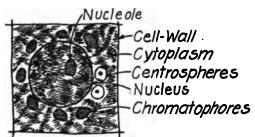


Fig. 3.—Plant-cell.

gen, oxygen, nitrogen, and sulphur combined in various proportions. Protoplasm has the power of imbibing or absorbing food which is dissolved in water. By absorbing food, protoplasm increases in size and when it reaches a certain size, it divides itself into two, four, or more parts. The newly formed cells absorb food and continue to grow; on reaching a certain size, they divide themselves again; they continue this process as long as When this process continues in a conditions are favorable. plant, it increases in size, that is, grows. Protoplasm also has the power to move. It has been observed that protoplasm changes its form, shifts the position of its several constituent parts, and in some cases moves its whole body from one place to another. The imbibition of food and the movements of the body of protoplasm take place at a temperature between 20° and 35°C, or 68° and 95°F.

The Chromatophores found in the green parts of plants and stained green by chlorophyl are called Chloroplasts or Chlorophyl Granules. Chlorophyl is a green coloring matter found in all plants. It is formed in the light only. The function of the chloroplasts is the formation of starch. Chromatophores. occurring in flowers. fruits. and some roots. carrot, and stained yellow or red with xanthophyl, Chromatophores, occurring in parts of called Chromoplasts. plants which are not exposed to the light, have no coloring matter and are called Leucoplasts. When exposed to the light, leucoplasts develop chlorophyl and become chloroplasts. When more food material than the protoplasm can assimilate is absorbed, it remains in the interior of the cells, in drops which are called Vacuoles. Starch grains are also found in plant-cells.

Water plays a very important rôle in the formation of plant-cells. It saturates the cell-walls, fills vacuoles holding food material in solution, and constitutes a greater part of the composition of protoplasm. The water performing this function is called *Cell-sap*. Cell-sap holds in solution many substances, most of which are plant-foods.

In higher forms of plants, many cells remain united and form tissues. As to the various kinds of tissues, the readers are referred to some text book on botany.

As was stated previously, protoplasm absorbs food, increases in size, multiplies in number, and causes a plant to grow. The following elements are what seem to be the food necessary for the growth of plants:— Carbon, hydrogen, oxygen, nitrogen, sulphur, iron, calcium, potassium, phosphorus, and silicon. Carbon is furnished by carbon dioxide in the air; hydrogen and oxygen, by water in the soil; calcium, by carbonate or sulphate of calcium; nitrogen, by ammonia and nitrates; sulphur, by sulphate; potassium, by sulphate or chloride of potassium; phosphorus, by phosphates; silicon, by silicates; iron, by iron oxide. All these substances, excepting carbon dioxide, are furnished by the soil in the form of water solution. They must remain dissolved in water so that they may be carried to different parts of a plant

by Osmotic Action or Diffusion and be absorbed and assimilated by protoplasms.

When a parchment paper sac containing a solution of sugar or salt is placed in water, the soluble substance diffuses out through the paper, while water diffuses in, until the strength of the solution inside and outside the sac becomes equalized. This is called Osmose. Speaking of a group of cells of a piant, when some cells contain more concentrated sap than others, osmotic action takes place between the cells until the concentration of cell-saps becomes uniform. The cells forming the parts of a plant exposed to the air and light contain more concentrated sap, owing to the evaporation and the formation of new substances, hence the soluble substances move from the leaves and stalks to the roots. On the other hand, the cells composing the roots and the parts not exposed to the air and light contain less concentrated sap, hence water passes upwards from the roots to the stalks and leaves, where it partly evaporates and partly combines with carbon dioxide to form starch.

Growth of the Sugar-Beet.—Any higher plant may be considered in the description of the formation of sugars and starch in plants and their functions in plant growth; but in this work, the growth of sugar-beets will be studied so that sugar-chemists may become familiar with some of their habits.

The sugar-beet is called, in botany, Beta vulgaris and belongs to the chenopodiaceae family of the subclass dicotoledoneae of the class angiospermae (Flowering plant). The shells of beet seeds occur usually agglomerated, that is, several seed shells stuck together, the number of seeds varying from one to seven. These agglomerations are commonly called Seed-balls. The greater number of seed-balls contain two or three seeds; some, only one seed. Each seed contains one embryo, that is, a rudimentary, undeveloped plantlet, which is composed of comparatively few cells, provided with food such as starch, fat, albumin, and so forth, for its growth.

When the seed is planted in the soil, supplied with moisture, and favored with the proper temperature, the food material

contained in the seed is rendered soluble by the influence of enzyme; and the protoplasms in the cells absorb the food and enlarge themselves; when they reach full size, they divide themselves in order to form new cells, continue this process, and multiply as long as conditions are favorable. Thus the plant grows. In growing, the plantlet first extends its root into the ground; the root is covered with hair roots by means of which the plant absorbs the moisture from the soil. When the plant takes a firm hold in the ground by its roots, it slips off its cotyledons, that is, the first pair of leaves, from the seed and commences upward growth. Before it reaches the surface, food is supplied from the seed and water, from the soil. After reaching the surface, it absorbs carbon dioxide from the air and manufactures its own food.

The absorption of carbon dioxide by plants from the air is accomplished by means of peculiar organs called *stomata* (singular, stoma-mouth). If we examine the epidermic tissue of the green part of any higher plant under a powerful microscope, we observe very irregular tissues among which stomata are abundantly scattered. A stoma consists of two cells bearing chlorophyl granules; between the two cells, there is an opening which penetrates through the epidermis into the interior intercellular spaces, that is, the spaces between the interior cells. Stomata are found on the leaves and stems and usually more abundantly on the lower surface of leaves. In the sunlight, air passes through these openings into the intercellular spaces where the interior cells absorb carbon dioxide, assimilate carbon, and set oxygen free. For this reason, the stomata are called the breathing pores of plants.

As soon as a beet plantlet reaches the surface, the chromatophores in the cells of the cotyledons become stained with chlorophyl, thus converting themselves into chloroplasts, preparatory to making starch. When air passes through the openings of stomata into the intercellular spaces, the cells surrounding these spaces absorb carbon dioxide gas, which the chloroplasts, by the influence of the sun light, cause to combine with the water which is already present in the cells, the result being the formation of starch. Just what reaction takes place during the formation of starch is not exactly known. It is, however, highly probable that such a series of chemical reactions take place as are discussed below. When carbon dioxide combines with water, carbonic acid (H<sub>2</sub>CO<sub>3</sub>) is formed. This acid may, in some manner, be converted into formaldehyde by simply setting a part of its oxygen free:

Formaldehyde thus formed may be converted into glucose by condensing six molecules thereof:

$$\begin{array}{lll} \text{Formaldehyde} & & \text{Glucose} \\ 6(\text{CH}_2\text{O}) & = & \text{C}_6\text{H}_{12}\text{O}_6 \end{array}$$

This glucose may be converted into starch by dehydrating or by subtracting one molecule of water therefrom:

Glucose Water Starch 
$$C_6H_{12}O_6$$
 —  $H_2O$  =  $C_6H_{10}O_5$ 

Whether these changes actually take place or not, it is experimentally proved that oxygen is set free during the formation of starch.

Starch is insoluble in water and could not be conveyed to different parts of a plant by osmotic action as insoluble substances could not diffuse through the cell-walls; hence, starch must be converted into some soluble forms. Usually it is transformed into glucose, sucrose, or inulin (soluble starch). The soluble carbohydrates thus formed are conveyed to all parts of a plant by osmotic action so that the cells may be constantly supplied with food material. As was stated before, the composition of protoplasm comprises, besides carbon, hydrogen and varied quantities of nitrogen, sulphur, phosphorus, potassium, calcium, and iron. These elements are supplied mainly from the soil in various forms of salts. The water which holds these salts in solution and surrounds the root of a beet is very dilute or very much less concentrated than the cell-sap in the root of the beet. hence these salts are, by osmotic action, readily absorbed into the cells in the roots together with water and thence carried to all parts of the plant body where they are assimilated by the protoplasms. This process goes on until the plant reaches full maturity or until it bears seeds, allows all the seeds to ripen, and dies.

In many plants, carbonaceous food material is stored away in certain parts of the plants; for example, in potatoes, starch is first formed in the leaves, transformed into soluble substances, and conveyed to the tubers, where it is converted into starch again by the action of leucoplasts and deposited for future use. In the case of the sugar-beet, starch formed in the leaves is transformed into soluble substances, conveyed to the roots, and stored there in the form of sucrose.

The sugar-beet was originally an annual plant, that is, the wild beet sprouts from seeds, grows, and bears seeds in one season, but the domesticated beet has become bi-annual, that is, it requires two seasons to reach full maturity after germination; during the first year of its growth, its entire effort is directed to the development of its root and the accumulation of sugar therein by means of its leaves and other necessary substances through its rootlets to be used in its future development. function, during the second season, is to complete the object of its existence:— the bearing of seeds. It first develops various stalks with alternate leaves and usually with branches, and then flowers between the stalk and the leaves on the various parts of the stalk. Under favorable conditions, these flowers produce matured seeds at the end of the season. As to the description of parts of the flowers and seeds and their functions, the readers are referred to some text book on botany. All these functions, during the second year, are performed largely at the expenditure of the food material,—sugar and so forth stored up in the root during the first year of its growth; hence, it is natural that the highest sugar content in a beet is reached at the end of the first season. This fact in the course of Nature's work has been discovered by man and taken advantage of in the manufacture of sugar. It is for this reason that beets for sugar extraction are harvested at the end of the first season

Composition of Beets.—Beets consist of marc and juice. Marc

includes fibres and other insoluble substances. Juice comprises water and all the substances dissolved therein. The proportion of these two constituents varies considerably according to conditions of so.l, climate, weather, and various other circumstances. Marc varies between 3 and 7 per cent and juice, between 93 and In ordinary practice, 5 per cent is taken for the 97 per cent. average of marc and 95 per cent, for juice. The knowledge of these factors is very important, owing to the fact that, in commercial practice, the sugar content is estimated in the juice expressed from beets and the sugar content in the beets is calculated by the use of the factors. Since the juice, when separated from the marc, is the beet deprived of 5 per cent of their non-saccharine constituent, it may be regarded, so far as sugar and soluble non-sugar contents are concerned, as the beet concentrated to 95 per cent of their total weight. It is evident, then, that the sugar content found in the juice is 5 per cent higher than that found in the beets by a direct analysis; hence, when sugar is estimated in the juice and is to be calculated in per cent to the weight of beets, 5 per cent should be subtracted from the sugar content in the juice; for example:

Since the juice may be regarded as the beets concentrated to 95 per cent of their original weight and the sugar content therein has been increased in inverse proportion to the concentration, the sugar content in beets may also be obtained by multiplying the sugar content in the juice by the juice factor 0.95; for example:

Sugar in juice	Juice			Sugar in beets	
per cent	factor			per cent	
15.7	×	0.95	=	14.915.	

The method for determination of marc will be described in the chapter on Special Analysis.

Purity of Beet-Juice.—As stated above, beet-juice consists of water and substances dissolved therein. This soluble substance is called solid matter or dry substance and may be divided into sugar and non-sugar. Non-sugar may be subdivided

into organic substances and mineral salts. The amount of total dry substance in beet-juice varies all the way from 10 to 25 per cent. Sugar makes up the greater part of the total dry substances in beet-juice. The percentage of sugar in the total dry substance of beet-juice is also very variable, ranging from 70 to 90 per cent. The balance of the dry substance ranging from 10 to 30 per cent is made up of non-sugars. The percentage of sugar in the total dry substance of beet-juice may be obtained by dividing the percentage of sugar in the juice by that of the total dry substance in the juice and multiplying the quotient by 100; for example, when we have 15.4 per cent of the total dry substance and 12.5 per cent of sugar in beet-juice, the percentage of sugar in the total dry substance would be:

$$\frac{12.5}{15.4} \times 100 = 81.17.$$

This percentage is called the Co-efficient of Purity, or simply, Purity. The quality of such a juice as that just referred to is expressed by saying that the purity co-efficient of the juice is 81.17 per cent. Of 100 pounds of dry substance obtained by evaporating such a juice to dryness, 81.17 pounds are sugar and the balance, 18.83 pounds, are non-sugars.

The purity of beet-juice is of great importance, from the commercial standpoint, as the extraction of sugar largely depends on purity. The higher the purity of the juice, the greater the extraction; the lower the purity, the less the extraction. Crystallizability of sugar ceases when the purity of the juice or syrup is reduced to 60 per cent, that is, when the ratio of sugar to non-sugar becomes 60 to 40, no more sugar can be obtained by the process of crystallization. Roughly speaking, 40 pounds of non-sugar prevents 60 pounds of sugar from crystallization. In other words, when one pound of non-sugar is present in a

juice or syrup, it will almost invariably hold back  $\frac{60}{40} = 1.5$ 

pounds of sugar in syrup. It is evident then that, from 100 pounds of dry substance whose purity is 80 per cent, we are able to obtain, by crystallization,  $80-(20\times1.5)=50$  pounds of sugar;

from 100 pounds of dry substance with 70 per cent purity,  $70-(30\times1.5) = 25$  pounds of sugar; from 100 pounds of dry substance with 60 per cent purity,  $60-(40\times1.5)=0$  pounds.

It is, however, said that not all non-sugars prevent crystallization of sugar. Those which crystallize out before sugar begins to crystallize do not prevent the crystallization of sugar. Sulphates of sodium and potassium are said to be such.

Constituents of Non-sugars in Beet-Juice.—A large part of non-sugars in beet-juice is removed during the treatment thereof with lime and what could not be removed remains in the juice and offers various difficulties to the crystallization of sugar. Since the knowledge of the properties of these substances will be of great value to sugar-experts, the most characteristic properties of those substances hitherto identified in beet-juice will be discussed briefly in this article.

The optical activity and the solubility of the salts of lead and calcium of these compounds will be specially mentioned whenever available. The optical activity and the solubility of the lead salts of these compounds are especially interesting from the analytical standpoint, for optical activity interferes with optical analysis of sugar and many of these compounds may be removed as insoluble lead salts before polarization. The solubility of the calcium salts of these compounds is also of great importance as the degree of purification of beet-juice largely depends on the amount of these non-sugars removed by the treatment with lime.

Non-sugars may be divided into Organic and Inorganic Compounds; Organic compounds, into Nitrogen-free and Nitrogenous Compounds; Nitrogen-free compounds, into Plant Acids, Gummy Substances, Saccharine Substances, Coloring Matter, Fat and Resin, and Aromatic Compounds.

Plant Acid.—Plant acid is said to be the oxidation product of starch.

- (1) Glycolic Acid, CH<sub>2</sub>(OH)—COOH.—Soluble in water, alcohol, and ether. Salts of calcium and lead, soluble in water.
  - (2) Glyoxalic Acid, CH(OH)2—COOH.—Soluble in water

and alcohol. When boiled with lime, splits itself into glycolic and oxalic acids. Calcium salt is soluble in water; lead salt is insoluble in water.

- COOH

  (3) Oxalic Acid, | .—Soluble in water, alcohol, and COOH

  ether. Calcium salt, insoluble in water but slightly soluble in sugar solution; lead salt, insoluble in water.
- (4) Malonic Acid, CH COOH

  COO

and ether. Calcium salt, soluble in boiling hot water; lead salt, insoluble in water.

- CH<sub>2</sub> COOH

  (5) Succinic Acid, | .—Difficultly soluble in cold CH<sub>2</sub> COOH

  but readily soluble in hot water or alcohol. Neutral calcium salt, difficultly soluble in water; lead salt, insoluble in water.
- (6) Glutaric Acid, CH, COOH
  .—Readily soluble in CH, COOH
  water, alcohol, and ether. Calcium salt, easily soluble in cold but difficultly soluble in hot water.
- (7) Adipic Acid, (CH<sub>2</sub>).—Soluble in hot water, alcohol, and ether. Calcium salt, difficultly soluble in hot and cold water.
- CH<sub>2</sub> COOH

  (8) Malic Acid, | .—Soluble in water and

   CH(OH) COOH

  alcohol. Neutral calcium salt and lead salt, insoluble in water.

  Levo-rotatory, specific rotation being  $[a]_D^{20} = -2.62^{\circ}$  with 5 per cent solution; in more concentrated solution, dextro-rotatory, the
- CH(OH) COOH

  (9) Tartaric Acid, | .—Readily soluble in CH(OH) COOH

  water and difficultly soluble in alcohol. Neutral calcium salt, difficultly soluble in water; lead salt, insoluble in water. Dextro-

specific rotation being  $[a]_{D}^{\infty} = +5.89^{\circ}$ .

rotatory, specific rotation being  $[a]_D^{*0} = +15.06^{\circ}$  in a solution containing from 0.5 to 15 grams in 100 c.c. water.

Soluble in water and alcohol, difficultly soluble in ether. Salts of calcium and lead, insoluble in water.

ularly, but rarely in beets. Neutral calcium salt, difficultly soluble in cold water, precipitates when boiled; lead salt. insoluble in water.

water and alcohol, difficultly soluble in ether. Calcium salt, soluble in water but separates when boiled; lead salt, insoluble in water.

alcohol, and ether. Calcium salt, gelatinous becoming crystalline when boiled; lead salt, insoluble in water.

Gummy Substances.—These substances were formerly known as Pectine Matters.

(1) Arabic Acid, C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>.—Formerly known as Meta-pectic Acid. A variety occurring in the sugar-beet is levo-rotatory, soluble in water, dissolves when boiled with milk of lime. Lead acetate does not precipitate. Basic calcium salt, insoluble in water.

- (2) Meta-arabin,  $(C_{12}H_{20}O_{10})_n$ .—When boiled with lime or other alkali, it dissolves and takes up one molecule of water, converting itself into arabic acid.
- (3) Pararabin, C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>.—Readily soluble in a weak acid; precipitates from solution with alkali or alcohol; forms insoluble compounds with barium oxide and lead oxide.
- (4)  $\gamma$ -Galactan,  $C_6H_{10}O_5$ .—Soluble in hot milk of lime; strongly dextro-rotatory, specific rotation being  $[a]_D^{\infty} = +238^{\circ}$  in a solution containing 10 grams in 100 c.c.
- (5) Dextrin, C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>, and Levulin, C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>.—Not found in the original composition of beets but sometimes found as the products of fermentation.

Saccharine Substances.—Besides cane-sugar, there are two saccharine substances in beet-juice which must be considered as non-sugars, since they act as disturbing elements in the process of sugar manufacture. (1) Raffinose or Melitriose,  $C_{18}H_{82}O_{16}$ .  $5H_2O$ . (2) Invert Sugar,  $C_6H_{12}O_6 + C_6H_{12}O_9$ . The properties of these two substances are described under Carbohydrates.

Coloring Matters.—The coloring matter in beets is known as Chromogen. Very little is known about these substances in beets. Beet-juice is originally colorless, but as soon as it comes in contact with air, it becomes oxidized and turns first red, then blue, and finally quite dark. Reinke calls the original compound Rhodogen and the oxidation product which turned red Betarot.

Fat and Resin.—Beets contain several fatty substances, of which two have been identified.

- (1) Phytosterin,  $C_{26}H_{44}O$ .—An alcohol belonging to cholesterine; insoluble in water, soluble in alcohol, ether, chloroform, and petroleum ether. Levo-rotatory, specific rotation being  $[a]_{D}^{20} = -34.2^{\circ}$  when observed in 1.636 per cent solution using chloroform as solvent.
- (2) Beet-resin Acid, C<sub>22</sub>H<sub>36</sub>O<sub>2</sub>+H<sub>2</sub>O.—Insoluble in water, soluble in boiling absolute alcohol; calcium salt, insoluble in water; dextro-rotatory.

Aromatic Compounds.—Some years one notices strong scent

of vanilla in raw sugar; this is said to be due to the presence of vanillin which gives the scent to vanilla.

sugar; readily soluble in alcohol and ether but difficultly soluble in water; reduces Fehling's solution.

(2) Coniferin, 
$$C_6H_3$$
 O- $C_8H_{11}O_5$  .—Regarded to be the  $C_8H_4OH$ 

mother-substance of vanillin; prepared from woody beets; levorotatory, specific rotation being  $[a]_D^\infty = -66.90^\circ$  when observed in 0.896 per cent solution; does not reduce Fehling's solution.

(3) Pyrocatechin, C,H, OH OH .—Found in raw sugar containing vanillin; reduces Fehling's solution.

Nitrogenous Compounds.—Several organic nitrogenous compounds have been identified by various authorities. They are the source of the development of ammonia in beet-juice.

(1) Leucine,  $C_4H_9CH(NH_2)COOH$ .—Easily soluble in hot water, difficultly soluble in cold alcohol, insoluble in ether; crystallizes from cold water; dextro-rotatory, specific rotation being  $[a]_D^\infty = +8.05^\circ$  when observed in a liquid containing 2.371 grams of the substance in 100 c.c. of 4 per cent sodium hydroxide solution.

water, insoluble in alcohol and ether. When boiled with dilute mineral acid or alkali, it splits itself into ammonia and aspartic acid  $(C_2H_3(NH_2) < {COOH \atop COOH})$ . Aspartic acid is soluble in hot water and insoluble in absolute alcohol. Asparagine and as-

partic acid are levo-rotatory in a water or alkaline solution and dextro-rotatory in an acid solution.

- (3) Glutamine,  $C_2H_6(NH_2) < \begin{array}{c} CONH_2 \\ COOH^2 \end{array}$ .—Fairly readily soluble in water but insoluble in absolute alcohol; optically inactive in a water solution, slightly dextro-rotatory in an acid solution. When boiled in a weak mineral acid or alkali solution, it splits itself, like asparagine, into ammonia and glutamic acid  $\left( \begin{array}{c} C_3H_5(NH_2) < \begin{array}{c} COOH \\ COOH \end{array} \right)$ . Glutamic acid is difficultly soluble in cold but readily soluble in hot water and insoluble in absolute alcohol and ether; it is dextro-rotatory in a water or acid solution, levo-rotatory in an alkaline solution.
- (4) Tyrosine,  $C_6H_4(OH).C_2H_3(NH_2)COOH$ .—Difficultly soluble in cold but readily soluble in hot water; insoluble in absolute alcohol and ether; dextro-rotatory, specific rotation being  $[a]_D = +6.85^{\circ}$ .

Leucin, aspartic acid, glutamic acid, and tyrosin are formed by boiling albumen with an alkali and also during the defecation of beet-juice with lime, indicating that beet-juice contains albuminous compounds.

(5) Albumen or Albuminate.—Occurs in all living plants, being the chief constituent of protoplasm. Its chemical structure is not known. It is composed of carbon, hydrogen, nitrogen, oxygen, and sulphur; the percentages of these elements vary according to various sources:

Carbon	52.7	to	54.5
Hydrogen	6.9	to	7.3
Oxygen	20.9	to	23.5
Nitrogen	15.4	to	16.5
Sulphur.	.8	to	2.0.

Albumen precipitates in an acid but dissolves in an alkali solution. Albumen obtained from an egg is levo-rotatory, its specific rotation being  $[a]_D = -37.8^{\circ}$  when observed in a dilute hydrochloric acid solution. Albumen contained in beets partly goes into the diffusion juice and suffers various decompositions during the purification of the juice. The albumen dissolved in the

juice may be partially coagulated by boiling; coagulation may be completed by adding a few drops of acetic acid. The first decomposition product of albumen during the extraction of beetjuice is peptone. Peptone diffuses through a membrane very readily while albumen does not. Peptone is levo-rotatory.

- (6) Lecithin, C<sub>42</sub>H<sub>84</sub>NO<sub>9</sub>P.—Waxy substance; soluble in alcohol and ether; when boiled with an acid or alkali, breaks up into cholin, glycerin, phosphoric acid, stearic and palmitic acids.
  - (7) Cholin,  $C_2H_4 \stackrel{\frown}{=} N \equiv (CH_8)_8$ .—Strongly basic syrup; read-OH

ily soluble in alcohol; not poisonous.

- (8) Betain,  $CH_2 \subset \binom{CO_2}{|N|}$ .—Readily soluble in water and alcohol.
- (9) Citrazinic Acid, C<sub>5</sub>H<sub>2</sub>N .—Yellowish crystalline powder; found in poorly conserved beets; soluble in alkali and ammonia; calcium salt, difficultly soluble in water; lead salt, insoluble in water.

Inorganic Substances.—Potassium, sodium, calcium, magnesium, and iron are found in beets, partly combined with organic acids and partly, with mineral acids, such as sulphuric acid, phosphoric acid, and chlorine. Nitrates and ammonia salts are also found in beets.

## Chapter VI.

## THE POLARISCOPE AND ITS ACCESSORIES.

This chapter will be devoted to the discussion of the apparatus necessary for the polarimetric determination of sugar.

Optics.—In order to understand the polariscope, it is necessary to know a few fundamental principles of Optics. The true nature of light is not exactly known; suffice it to say here that light is one of the phenomena of energy which excites the sensation of

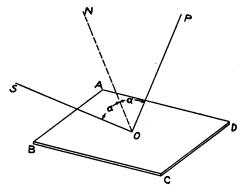


Fig. 4.—Diagram showing reflection of ray of light.

vision on the retina of the eye. Optics treats of the properties of light. Of its numerous properties, the laws of reflection, polarization, and refraction are most important for the study of the polariscope.

Reflection of Light.—Any line perpendicular to a plane surface is called normal; for example, referring to Fig. 4, the line N O is called normal when it is perpendicular to the plane A B C D. When a ray of light, coming from the direction S O, falls upon the point O, S O is called an incident ray; the angle, formed between the incident ray and the normal or S O N, the angle of incidence; the reflection, cast in the direction O P, the reflected ray; the angle between the normal and the

reflected ray or N O P, the angle of reflection. The angle of reflection is always equal to the angle of incidence.

Polarization of Light.—In Fig. 5, let N O be a normal to the unsilvered glass plate A B C D, S, a source of light, S O, the incident ray and O P, the reflected ray; when S O falls on the plate A B C D at the point O, making an angle of 54.35° with the normal N O, the light, reflected in the direction O P, becomes completely polarized; at other angles, partially polarized. It is

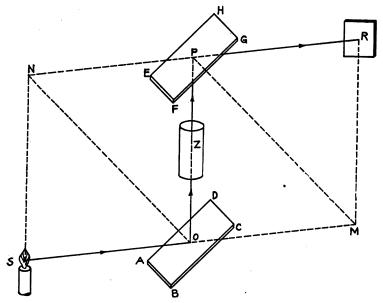


Fig. 5.—Diagram showing polarization of ray of light by reflection and plane of polarized light.

called polarized because of its disposition to exhibit opposite properties under different conditions, as if influenced by poles. The waves of polarized light vibrate in a single direction, while those of unpolarized light move in all directions.

Property of Polarized Light.—Let the reflected ray P O (Fig. 5) fall at the point P on the second glass plate E F G H, arranged exactly parallel to the first plate, then the ray will again be reflected in the direction P R, forming a bright spot R

on the screen. In reference to the second plate, P M is the normal; PO, the incident ray; PR, the reflected ray; OPM, the angle of incidence, and M P R, the angle of reflection. Now imagine two planes:—one containing O S, O N, and O P or O S N P, the other containing P O, P M, and P R or P O M R. As long as the two glass plates remain exactly parallel and the two imaginary planes O S N P and P O M R are lying in the same plane, that is, the angle formed between the two planes remains at 180°, the intensity of the reflected ray of polarized light is the same as that of ordinary or unpolarized light. the plate E F G H turn round O P as an axis without changing the angle of reflection M P R; the intensity of the reflected light begins to diminish and continues to do so until it turns 90°, when no light will be reflected. In other words, when the imaginary plane P O M R is turned until it is at right angles to the plane O S N P, there will be no reflection of light on the screen R. When the plate E F G H is turned past this point, the light begins to reflect and increase the intensity of the reflected ray until it reaches its maximum, when the imaginary planes O S N P and P O M R coincide. At this point, the intensity of the reflected light is the same as it was at the opposite or original position. When the plate E F G H is turned further, the intensity of the reflected ray begins to diminish again and continues to do so until the planes POMR and OSNP form a right angle at the opposite side, when no light is reflected. When the plate is turned past this position, the light again reflects and increases the intensity until it reaches the original position. The revolution of the plate E F G H in the opposite direction will produce exactly the same effects. It is evident from these facts that whenever the planes O S N P and P O M R are in one plane, or parallel to each other, the light is reflected with the same intensity as that of unpolarized light, that whenever they are forming a right angle, or perpendicular to each other, no ray is reflected, and that at other positions, the reflection is variable, according to the dimensions of the angles formed between them. The plane in the direction of which ether vibrates and the maximum is transmitted is called the Plane of Polarized Light. When light is polarized by reflection, the plane in which the maximum ray of polarized light is reflected is called the plane of polarized light, therefore the plane O S N P may be called the plane of polarized light.

Rotation of Plane of Polarized Light.—There are many substances which have a peculiar action toward polarized light; under certain conditions, they rotate the plane of polarized light. This is very important, as the mechanism of the polariscopes and saccharimeters is based on this behavior of substances. Of many optically active substances, cane-sugar will be taken up here for studying the rotation of the plane of polarized light, as the object of this article is to lead up to the saccharimeter, which is a special type of polariscope.

Let E F G H (Fig. 5) be revolved until the planes O S N P and P O M R form a right angle and there will be no reflection on the screen R; let a glass or metal tube Z be filled with a sugar solution, closed at both ends with glass plates and metal screw-caps and be so interposed between O and P that the reflected ray may pass through the solution; then the screen will instantly be brightened, indicating a reflection of light. In order to restore to the position where there is no reflection of light, the plate E F G H must be turned to the right or in the direction of a watch-hand through certain degrees. From this observation, we are able to assume that the sugar solution has rotated the plane of polarized light O S N P (Fig. 5) in such a manner as indicated by Fig. 6 in which (a) shows the original plane and (b), the one rotated by the sugar solution.

Other things being equal, the degree of rotation varies according to the strength of the solution and the length of the column of solution; the stronger the solution, the greater the rotation; the longer the column, the greater the rotation.

Polarization of Light by Iceland Spar Prism.—The arrangement shown in Fig. 5 is very convenient for a study of the principles of the polarization of light and the behavior of optically active substances on the polarized light. Such an arrangement, however, could not be applied for accurate observations, owing

to its comparatively less complete polarization and the difficulties in constructing a compact and accurate instrument based on this principle. Fortunately, it has been discovered that when a ray of light passes through a prism of Iceland spar, it suffers a double refraction, that both rays become completely polarized and that the pianes of polarization of the two rays are perpendicular to each other. Most polariscopes used in scientific as well as in commercial work are made with Iceland spar prisms.

Single Refraction of a Ray of Light.—In order to understand the construction of polariscopes made with Iceland spar prisms,

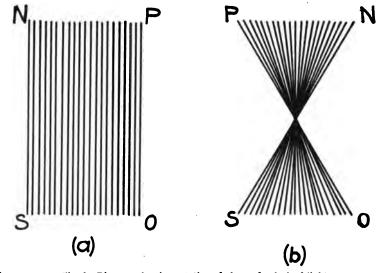


Fig. 6.—Diagram showing rotation of plane of polarized light.

it is very necessary to know the fundamental principles of single and double refraction. When a ray of light passes from one medium to another of different density, perpendicularly, it suffers no deflection, or, it does not bend. This perpendicular line is called *normal* and is indicated by A B (Fig. 7). When a ray passes obliquely, it changes its course at the point O on the separating line M N as shown by C O D. The angle formed between the normal and the incident ray, that is, A O C, is called the Angle of Incidence; the angle between the normal and the

refracted ray, that is, D O B, the Angle of Refraction. When the density of the medium below M N is greater than that of the one above M N, the refracted ray bends toward the normal, that is, the angle of refraction is smaller than that of incidence. When, on the contrary, the density of the medium below M N is lower than that of the one above M N, the refracted ray bends away from the normal, that is, the angle of refraction is greater than that of incidence; for example, suppose the medium above M N to be air and that below M N, pure water, the ray O D will bend toward the normal O B; on the contrary, should the

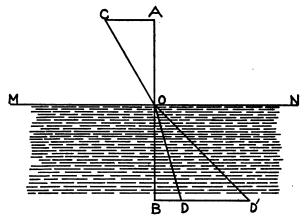


Fig. 7.—Diagram showing refraction of ray of light.

medium above M N be solid glass instead of air, the ray will bend away from the normal as shown by O D'.

inch. The refractive index between the air and the water would, then, be  $\frac{A}{B}\frac{C}{D} = \frac{1.336}{I} = 1.336$ . If we divide this result by the refractive index of air 1.000294, we obtain the absolute refractive index of water, that is, the refractive index obtained when a ray passes from a vacuum to water. The refractive index of air, however, is very small and may be disregarded for ordinary purposes. Should the medium above M N be air and that below M N be glass, C A would be 1.6 inches and B D, 1 inch; then the refractive index of glass would be  $\frac{1.6}{I} = 1.6$ . However great the obliquity of the incident ray C O or the angle A O C may be, this ratio or refractive index is al-

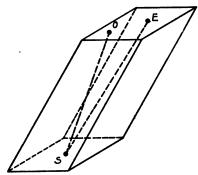


Fig. 8.—Diagram showing double refraction of ray through Iceland spar prism.

ways the same. These phenomena are observed in uncrystallized media such as air, liquids, ordinary glass, and the like.

Double Refraction of Ray of Light in Iceland Spar.—There are certain crystallized bodies in which a ray is doubly refracted or the refracted ray takes two courses with equal intensity as shown by O D and O D' (Fig. 7). This phenomenon is called Double Refraction or Bifurcation. The most prominent one among this class of crystals is the Iceland Spar Prism. If we make a dot S (Fig. 8) on a white paper and observe it through a crystal of Iceland spar, we will observe images of two dots. This is due to the fact that a ray entering the crystal takes two courses and emerges at different points as indicated by S O and S E (Fig.

8.) S O is called the *ordinary ray* and S E, the *extraordinary ray*. Should the crystal be turned with the face still on the paper, the ordinary image will remain fixed while the extraordinary image will describe a circle around it. The refractive index of the ordinary ray is 1.654 and follows the laws of single refraction, that is, the index is constant. The refractive index of the extraordinary ray is 1.483 and does not follow the laws of single refraction, that is, the index is variable according to the obliquity of the incident ray.

Polarization of Ordinary and Extraordinary Rays.—Ordinary and extraordinary rays possess other peculiar properties; they are polarized. The ordinary ray is polarized in the direction of the principal plane of the crystal and the extraordinary ray in the direction of a plane perpendicular to the principal plane.

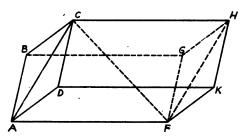


Fig. 9.—Diagram showing principal plane of Iceland spar prism.

The crystal form of Iceland spar as shown in Fig. 9 is a rhombohedron and has eight solid angles or corners, of which, two corners C and F are contained by three obtuse angles of 105.5°. A straight line connecting these two corners, C and F or any line parallel to this is called an Axis of the crystal. When a dot is observed through the crystal along the direction of this line, the image does not appear double; for this reason, it is called the Optical Axis. A plane which is drawn in such a manner that it includes the principal axis C F and is perpendicular to the faces A B C D and F G H K, that is, the plane A C H F is called the Principal Plane. Any plane parallel to this plane is also called a principal plane.

Planes of Polarization of Ordinary and Extraordinary Rays.—As stated above, the ordinary ray is polarized in the direction of the principal plane, that is, the plane A C H F, and the extraordinary ray, in the direction of a plane perpendicular to the principal plane. This may be explained by using two prisms of Iceland spar as shown in Figs. 10 and 11. When a ray from a certain

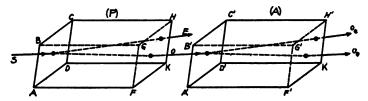


Fig. 10.—Diagram showing ordinary-ordinary and ordinary-extraordinary rays.

source S is allowed to pass through the first prism P (Fig. 10), it suffers double refraction and emerges at two points as indicated by O (Ordinary) and E (Extraordinary). Should the ordinary ray O be allowed to pass through a second crystal A, it suffers double refraction again and emerges at two points as indicated by O o (Ordinary-ordinary) and O e (Ordinary-extraordinary). When the crystals P and A are arranged in a similar position or A is revolved through 180°, that is, when the principal planes of A and P are in one plane or parallel to each other, only O o appears at its maximum intensity. When A is turned in either direction without changing the direction of the axis of vision,

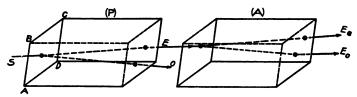


Fig. II.--Diagram showing extraordinary-ordinary and extraordinary-extraordinary rays.

O o begins to diminish and O e appears and increases its intensity with rotation; they continue to do so until A is rotated through 90°, when O e disappears entirely and O e attains its maximum intensity. When A is turned past this point, O e begins to diminish and O e appears and increases its intensity with rotation; they

continue to do so until A is revolved through 180° from the original position, when O o attains its highest intensity and O e disappears entirely.

The principal plane of the first prism P corresponds to the plane O S N P (Fig. 5) and that of the second prism A, to the plane P O M R. As stated previously, the plane of polarization is that plane in which ether vibrates and the maximum ray of Now Oo (Ordinary-ordinary polarized light is transmitted. ray) is transmitted through A at its maximum intensity when the principal planes of P and A are in the same plane or parallel, just the same as the polarized light is reflected at its highest intensity when O S N P and P O M R are in the same plane. This fact indicates that the ordinary ray is polarized in the direction of the principal plane of Iceland spar. On the other hand, Oe (Ordinary-extraordinary ray) is transmitted at maximum intensity when the principal plane of A is perpendicular to that of P, that is, when a plane in A perpendicular to the principal plane of A and the principal plane of P are in the same plane. This phenomenon affirms that the extraordinary ray is polarized in the direction of a plane perpendicular to the principal plane.

Should the extraordinary ray E from the first prism P be allowed to pass through the second prism A as shown in Fig. 11, the ray suffers double refraction again and emerges at two points, as indicated by Eo (Extraordinary-ordinary) and Ee (Extraordinary- extraordinary). Eo attains its maximum intensity when the prism A is revolved through 90° so that the principal plane of A is perpendicular to that of P or, a plane in A perpendicular to the principal plane of A and the principal plane of P are in the same plane or parallel, but it disappears entirely when the principal plane of A and that of P are in one plane or parallel. It is evident from this observation that the extraordinary ray is polarized through P in the direction of a plane perpendicular to its principal plane and, in consequence, the maximum transmission of the extraordinary-ordinary ray takes place when this plan coincides with or is parallel to the principal plane of the second prism A. On the other hand,  $\mathbf{E}e$  appears at its

maximum when the two prisms are arranged in similar attitudes and disappears entirely when the second prism A is turned through 90°. These observations confirm the assumption that the extraordinary ray is polarized through P in the direction of a plane perpendicular to its principal plane, and the maximum transmission through A takes place when that plane is in the same plane with or parallel to a plane in A perpendicular to the principal plane of A. Ee is the most important ray in polarimetric work, as it is employed in the observation of the rotation of the plane of polarized light, and the plane in which this ray is polarized will be called, hereafter, the Plane of Polarization or the Polarization Plane.

Elimination of Ordinary Ray.—In order to make the extra-

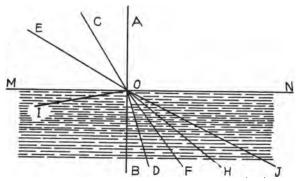


Fig. 12.—Diagram showing critical angle.

ordinary ray applicable in polarimetric observation, the ordinary ray must be eliminated, as the two rays act in opposite directions, that is, where one exhibits the maximum intensity, the other shows the minimum, and in consequence thereof, an accurate observation could not be made. The elimination is accomplished by the application of the law of total reflection. It has been stated previously that when a ray of light is allowed to pass obliquely from a certain refractive medium to a less refractive one, its course bends away from the normal. When the obliquity is increased, however, the ray ceases to emerge above the separating line at a certain degree. Fig. 12 illustrates the

case in which the medium above M N is air and that below M N, water; a ray D O diverges in the direction of O C; F O, in the direction of O E. At a certain degree of obliquity of the incident ray such as indicated by H O, the refracted ray coincides with the separating line M N and no ray emerges above the surface; hence, the angle of refraction is 90° while the angle of incidence is less than 90°. Such an angle of incidence is called the *Critical Angle*. When the obliquity of incident ray is increased, as shown by O J, the entire ray is reflected in the direction O I. When the media are water and air, the critical angle is about 48°, 30'; for Canada balsam and Iceland

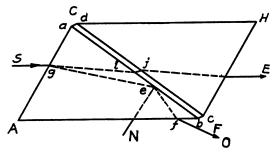


Fig. 13.—Diagram showing elimination of ordinary ray from Iceland spar prism.

spar, as regards the ordinary ray, the critical angle is about 68°, 55'.

In eliminating the ordinary ray, a rhombohedral prism of Iceland spar is bisected along the principal axis C F and perpendicularly to the principal plane A C H F (Figs. 9 and 13) and the two halves are cemented together by means of a layer of Canada balsam as shown by  $a \ b \ c \ d$  in Fig. 13. The refractive index of the ordinary ray is 1.654 and is higher than that of Canada balsam, which is 1.549. The prism is made long enough so that the incident angle  $g \ e$  N will be greater than the critical angle which is 68°, 55'; consequently, when the ordinary ray indicated by S  $g \ e$  (Fig. 13) falls upon the separating line  $a \ b$  at e, total reflection takes place and the ray emerges in the direction  $e \ f$  O. On the other hand, the extraordinary ray, whose refractive index is 1.483 and lower than that of

Canada balsam, on meeting a layer of Canada balsam refracts in the direction i j and follows the course indicated by j E, thus passing through the entire length of the prism. The prism thus prepared is called a *Nicol Prism* or simply a "*Nicol.*"

Variation of Intensity of a Polarized Ray Transmitted Through Two Nicol Prisms Arranged in Different Attitudes.— When the first Nicol, into which a ray enters and becomes polarizdesignated by P and the second, by A, and A when P are arranged in such a manner planes ofpolarization are in one plane parallel, a polarized ray passes through two prisms its maximum intensity; should A be turned in either direction,

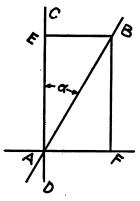


Fig. 14.—Graphic representation of components of plane of polarized light.

the brilliancy begins to diminish and continues to do so until it is rotated through 90°, when a complete darkness takes place. The variation of intensity may be best explained by the aid of a graphic representation (Fig. 14): let A B designate the plane of polarization of P, A F, that of A, and C D, the principal plane of A. When the two Nicols are so arranged that A B and A F would coincide, the maximum ray is transmitted; on the other hand, when A B coincides with C D, no ray is transmitted through A. When P is turned to the right, there will be formed an angle a between A B and C D; the intensity of the

transmitted ray increases with this angle in direct proportion until A B reaches A F.

In terms of higher mathematics, it may be said that the intensity of a ray polarized in the direction A B may, like force, be decomposed into two components A E and A F. A E which is equal to A B cos a is entirely extinguished by A since it coincides with the principal plane C D of A; on the other hand, A F which is equal to A B sin a is completely transmitted through A, since it coincides with the plane of polarization of A.

Observation of the Rotation of the Plane of Polarized Light.— In observing the rotation of the plane of polarized light, the two Nicols P and A are placed at some distance in such a manner that the plane of polarization of A is perpendicular to that of P and no ray is transmitted. This position may be called the zero-point. If we now interpose a tube filled with a cane-sugar solution between the two prisms, the ray will be transmitted in-In order to bring the field of vision to complete darkness. A must be rotated in the direction of the hands of a watch through certain degrees in direct proportion to the strength of It is evident, then, that the plane of polarized light the solution. has been rotated by the influence of sugar solution, in the manner shown by (b) in Fig. 6. The first Nicol P is called a Polarizer, as it polarizes a ray of light; the second Nicol A, an Analyzer. as it indicates in what direction a ray is polarized and how many degrees a polarized ray is rotated through a solution of cane-sugar or other optically active substances. The tube used for observing the rotatory power of solutions is called an Observation Tube.

Polariscope and its Use.—Fig. 15 illustrates an arrangement of one of the simplest types of apparatus used for observing the rotation of the plane of polarized light. Such an apparatus is called a Polariscope. P and A are the polarizer and the analyzer, respectively. Both are enclosed in open brass tubes. T is a brass trough for receiving the observation tube O and is provided with a cover C; g, a graduated circular scale; p, a pointer, attached to the tube holding the analyzer, for reading

the rotation of the analyzer; L, a lever for turning the analyzer. When the analyzer is adjusted by means of the lever until greatest darkness is reached in the field of vision, the pointer should rest exactly at the zero-point on the scale. When the analyzer is rotated in either direction, a ray begins to pass, increases its intensity, and reaches the greatest brightness the instant the analyzer is turned exactly through 90°; when the analyzer is turned past this point, light begins to diminish, and reaches the greatest darkness at 180°; when the analyzer is turned still farther, the greatest brightness is again observed at 270°; when it is turned past this point, the ray diminishes until it reaches the maximum darkness at the zero-point. It is evident, then, that

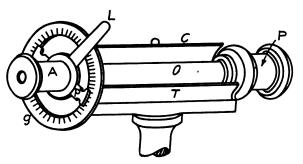


Fig. 15.-Polariscope with circular scale.

the apparatus has two zero-points, 180° apart. As regards the direction of rotation, it is said to have rotated to the *right* when the analyzer is turned in the direction of watch-hands and to the *left*, when turned in the opposite direction.

In observing the optical activity of cane-sugar, after having the polariscope turned toward a source of light and the analyzer set on the zero-point, fill the observation tube with a cane-sugar solution of certain strength and place in the trough, the field of vision will then instantly be brightened; in order to reach the darkest point, the analyzer must be turned in the direction of watch-hands; for this reason, cane-sugar is said to rotate the plane of polarized light to the right or to be dextro-rotatory (dextro=right). On the other hand, when we observe a fruit-

sugar solution, the analyzer must be turned to the opposite direction, and therefore, fruit-sugar is said to rotate the plane of polarized light to the left or to be *levo-rotatory* (levo=left). Any substance possessing such a property toward polarized light as just cited is called an *optically active body*.

The above is the general principle of the polariscope and the description of its essential parts. Instruments of this type may be used for all optically active bodies.

Requirements of a Good Polariscope.—In addition to the parts cited above, all polariscopes used for accurate observations are provided, as shown in Fig. 16, with an illumination lens, L, a polarizer diaphragm, D', an analyzer diaphragm, D', and a telescope composed of an objective, T, and an ocular, O. A straight line connecting the centers of the two diaphragms is taken as the

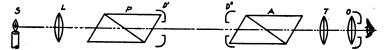


Fig. 16.—Essential optical parts of polariscope.

axis of the instrument. Nicol prisms are so placed that their principal planes run exactly parallel to this axis; the illumination lens and the telescope are also so arranged that their axes coincide with this line.

Principle of Half-Shadow Polarizer.—The disadvantage of a polariscope whose zero-point is located at total darkness is that the total darkness approaches and disappears very gradually and is very difficult to be determined exactly. This difficulty is overcome by the use of a half-shadow polarizer. The zero-point of a polariscope with a half-shadow polarizer is located in a partial shadow (commonly called half-shadow). The field of vision is divided into two, three, or four parts, which are called double, triple, or quadruple field, respectively. When the analyzer of a polariscope of this type is set on the zero-point, the whole field appears uniformly illumined. When the analyzer is turned slightly off the zero-point, some part of the field becomes brighter, while the other turns darker; either their bright-

ness or darkness increases on further rotation of the analyzer. Partial-shadow or half-shadow and division of the field are produced by a simple ingenious arrangement of Nicol prisms in constructing the polarizer. Let it be assumed that the field of vision of a polarizer A B C D (Fig. 17) is divided into two parts, as indicated by A E F D and E B C F and also that the polarization-plane of the ray coming from A E F D lies in the direction G H and that of the ray coming from E B C F, in the direction G J; the angle H G J is usually made a little less than 10° and is designated by a. Now let the analyzer be so placed in front of the polarizer that its principal plane lies in the direction G K, then G H is divided into two component rays,

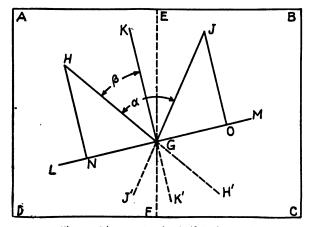


Fig. 17.—Diagram showing half-shadow angle.

G N and H N. The component ray H N, being parallel to G K, is completely extinguished by the analyzer, while the component ray G N, being perpendicular to G K, is entirely transmitted through the analyzer. G N, also being equal to G H sin  $\beta$ , varies with the angle  $\beta$  in direct proportion. When G K coincides with G H, the component ray G N becomes zero, consequently no ray will be transmitted through the analyzer and the field A E F D will appear completely dark. The same principle will nold true with the field E B C F. G J is divided into two component rays G O and O J. O J, being parallel to G K, is completely ex-

tinguished by the analyzer, while G O, being perpendicular to G K, is entirely transmitted through the analyzer. G O, also being equal to G J sin  $(a-\beta)$ , varies with the angle  $(a-\beta)$  in direct proportion. When G K coincides with G J, G O is zero. consequently no ray will pass through the analyzer and the field E B C F will be completely dark. From these observations, we may be able to understand that, when the angle  $\beta$  is equal to the angle  $(a-\beta)$ , or when G K bisects the angle a, the amount of light coming from the field A E F D will be equal to that coming from the field E B C F and both the fields will appear uniformly This point is called the optical equilibrium, or zero-Should the analyzer be turned to the right, the angle  $\beta$ enlarges while the angle  $(a-\beta)$  diminishes and consequently the field A E F D becomes brighter and E B C F, darker. the analyzer be turned to the left, the phenomenon would be reversed. When the analyzer is revolved through 180° from the position just described, the position of its principal plane relative to the two polarization planes G H and G J will remain the same as before, as may be seen from the relative positions of the planes G K', G H', and G J', which are simply extended planes of G K, G H, and G J respectively; hence, exactly the same phenomena would be observed. When the analyzer is revolved through 90°, that is, when its principal plane bisects the angles H G J' and J G H', the fields A E F D and E F C D will appear uniformly illuminated; this, however, could not be used for the zero-point of the polariscope; for the field of vision is too bright, owing to the too large angle of shadow and a slight difference in shade could not be distinguished.

Construction of Jellett's Half-Shadow Polariscope.—Jellett constructed the first half-shadow polariscope in 1860. He accomplished this by introducing, between the polarizer and the analyzer, an Iceland spar prism of special construction. An Iceland spar prism is first made a right prism by grinding off its ends and then is sawed through lengthwise, along a plane which makes the angle  $\frac{a}{2}$  with the plane of polarization and is put together in a reversed position, so that the directions of the plane of

polarization of the new prism will produce the half-shadow angle a. This is illustrated by (a) and (b) in Fig. 18, which represents one end of the prism. Let A C in (a) be a line indicating the direction of a plane of polarization and draw H E, a line indicating the direction of a plane which meets the plane of polarization A C at O, making the angle  $\frac{a}{2}$ . If we cut the prism along H E and then put the two halves together in a reversed position as shown by (b), the directions of the planes of polarization in the two halves of the prism would no longer be parallel to each other but make the angle a. The prism thus constructed is called a Half-Shadow Prism. It is enclosed in a

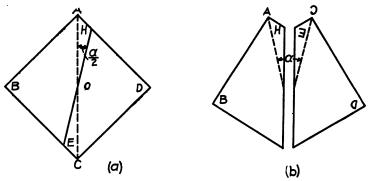


Fig. 18.—Diagram showing direction of polarization plane in half-shadow prism.

brass tube closed at both ends with diaphragms. The diaphragms are provided with circular openings at the center so that the field of vision will appear circular.

In a polariscope, this half-shadow prism is placed next to the polarizer, keeping the cut vertical; the polarizer is so placed that its principal plane makes a right angle with the cut of the half-shadow prism, or bisects the acute angles produced by the principal planes of the half-shadow prism; the analyzer is rotated until its principal plane is perpendicular to that of polarizer, or bisects the angle  $\alpha$  of the half-shadow prism. In this position, the field of vision will appear uniformly shaded. The positions of these constituents are arranged in the order shown in Fig. 19, in which

P represents the polarizer; H, the half-shadow prism; T, the observation tube; A, the analyzer. Should an optically active substance be placed in the observation tube, the uniform shade will disappear instantly; in order to regain the uniformly shaded position, the analyzer must be rotated through certain degrees either to the right or to the left, according to dextro- or levorotatory activity of substance observed.

A prism known as a Schmidt-Haensch polarizer is a modification of Jellet's. In this polarizer, before the two halves of the Nicol prism are cemented together, one of the halves is split along the plane C H F (Fig. 9), and a wedge with the angle a is cut out and then the three pieces are cemented together. This is called a *Twin Prism*. The prism thus constructed is placed in the usual position for the polarizer, keeping the cut just described toward the analyzer and vertical. In such a polarizer, the direction of the plane of polarization of one half makes the

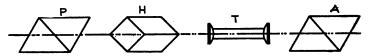


Fig. 19.—Positions of essential parts of half-shadow double-field polariscope.

angle a with that of the other half; hence, whenever, the analyzer is so placed that its principal plane bisects this angle, the field will be uniformly shaded.

Lippich's Half-Shadow Polariscope.—Lippich constructed the double-field and triple-field polariscope by a very simple arrangement of ordinary Nicol prisms. Only the arrangement of the polarizer will be described here, as the other optical parts are essentially the same as those of other polariscopes.

Lippich Double-field Polarizer.—In this polarizer, as shown in Fig. 20, a small Nicol is so placed in front of the polarizing Nicol that the former covers half the latter, the edge indicated by C dividing the field of vision vertically at the middle. It is for this reason that the smaller Nicol is called a half-prism. The polarizer is turned until its plane of polarization makes the angle  $\alpha$  with that of the half prism. As was explained previous-

ly, when a ray which is polarized in a Nicol prism passes through a second prism, whose principal plane is not parallel to that of the first prism, the ray is divided into two components and the second prism transmits only that component which is perpendicular to its principal plane; in consequence thereof, the ray coming through the half-prism is weaker than that coming from the free side of the polarizer and that part of the field of vision covered by the half prism will appear darker when the analyzer is so placed that its principal plane bisects the angle a. In order to obtain a uniformly shaded field of vision, the analyzer must be turned so far that the angle formed between the principal plane of the analyzer and the polarization plane of the half-prism is made so proportionately larger than that between the principal plane of the analyzer and the polarization plane of the polarizer as to compensate the diminished intensity. Polariscopes provided

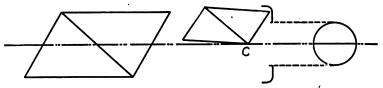


Fig. 20.—Lippich half-shadow double-field polarizer.

with this polarizer offer exactly the same phenomena as Jellett's when optically active substances are examined in them.

Lippich Triple-field Polarizer.—In this polarizer, as shown in the accompanying diagram (Fig. 21), two half-prisms B and C of equal size are so placed that they cover the polarizer P, leaving a free space between them, thus dividing the field of vision vertically into three parts whose areas are nearly equal to each other. The two half-prisms are arranged in symmetrical positions, that is, the position of one differs from that of the other by  $180^{\circ}$  so that their polarization planes remain in the same direction. The polarizer P is turned until its polarization plane makes the half-shadow angle a with those of the two half-prisms; the rays transmitted by the two half-prisms are equally diminished; hence the parts of the field of vision covered by the half-prisms would be equally shaded. When the analyzer

is turned until its principal plane divides the angle a in such a proportion as to compensate the diminished intensity of the ray through the half-prisms, the whole field of vision will be uniformly shaded.

The Necessity of using a Homogeneous Ray for Observation.— The rotation of the plane of polarization varies according to the wave-length of rays of different colors; the longer the wave-length, the smaller the rotation; the shorter the wave-length, the greater the rotation; hence, the rotation is least for the red ray and greatest for the violet ray. This is called dispersion of rays and varies for different substances. It is necessary for this reason to employ one homogeneous ray for polariscopic observa-

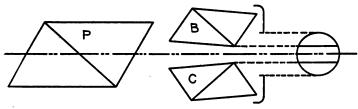


Fig. 21.—Triple-field polarizer.

tion. Any homogeneous ray would answer to this purpose but it is a common practice to employ the yellow ray produced by sodium compounds and known as  $Fraunhofer\ line\ D$ .

Precautions to be taken in Polariscopic Observation.—The room should be partly darkened, in order to keep the outside light from the instrument. The sodium light is placed at such a distance from the polariscope that it is focused on the polarizer diaphragm through the illumination lens. The telescope is focused sharply on the polarizer diaphragm. The zero-point must be determined accurately by rotating the analyzer from the right and left several times before observing active bodies. After the observation tube is filled with a solution of an optically active substance, the telescope must be focused again, as solutions change the focal length. Now the observer should slowly rotate the analyzer to the right or left, as the case requires, until the field of vision appears uniformly shaded and read the scale to tenths, using the vernier.

Specific Rotation of Solid Active Bodies.—Some crystals possess optical activity; the most commonly known is quartz. A section of quartz crystal, when cut perpendicularly to its axis, rotates the plane of polarized light. There are two kinds of quartz as regards optical activity; one is dextro-rotatory, the other, levo-rotatory. In such bodies, the angle of rotation produced by their optical activity varies with the thickness of the plate. Their rotating power is expressed by the angle produced by a plate one millimeter in thickness; for example, a quartz plate one millimeter thick, when observed at 20° C and with the ray D, rotates the plane of polarized light 21.7° to the right or left according to dextro or levo variety of the crystal.

Specific Rotation of Liquid Active Bodies.—The optical activity of a substance dissolved in some solvent or liquid carbon compounds is expressed by the angle rotated by a certain quantity of substance contained in a certain volume and observed through a certain length of column of the liquid. Such an angle is called the Specific Rotation and is usually represented by the symbol [a]. Specific rotation is, according to the universally adopted definition, that angle of rotation of a plane of a polarized ray produced by passing through a column of a liquid, one decimeter in length, which contains one gram of active substance in one cubic centimeter.

As the angle of rotation is variable with the wave-length of the ray employed and the temperature of the liquid examined, the ray and the temperature, with which the observation is made, are always expressed by a symbol. Observations are usually made with the yellow ray, the Fraunhofer line D and at 20°C. The specific rotation, observed under these conditions, is represented by the symbol  $[a]_D^m$ . When any other ray or temperature are employed, the rotation is expressed by a corresponding symbol, as for example  $[a]_D^{n_2}$ .

According to the definition given above, the specific rotation of an active liquid substance may be calculated from the formula:

I. 
$$[a] = \frac{a}{l.d}$$
,

in which a represents the angle of rotation in circular degrees, observed for a definite color; l, the length of the observation tube; d, the density or specific gravity of the liquid at a temperature t. When an observation is made with the ray D and at 20° C, the specific rotation is a characteristic constant; for example, the specific rotation of nicotine is  $\lceil a \rceil_D^2 = -161.55^\circ$ .

The specific rotation of a solid active substance dissolved in an inactive solvent may be calculated from the formula:

II. 
$$[a] = \frac{100 a}{l.c}$$
,

in which c represents a concentration or the amount of a substance in grams dissolved in 100 c. c. of a solvent. Example: for

cane-sugar, when 
$$a=34.68^{\circ}$$
,  $l=2$ ,  $c=26.048$ ,  $[a]=\frac{100\times34.68}{2\times26.048}=66.57^{\circ}$ .

When a weighed quantity of an active substance is dissolved in a weighed amount of an inactive solvent, the percentage of the active substance p, and that of the inactive solvent q may be easily obtained by a simple calculation. The density or specific gravity of this solution may be determined by means of a

pyknometer, using the formula, 
$$d_4^{20} = \left(\frac{F}{W} \times 0.99705\right)$$
—0.00120,

in which W represents the weight of water in grams at  $20^{\circ}$ C; F, that of the liquid at  $20^{\circ}$ C; 0.99705, the density of water at  $20^{\circ}$ C referring to the density of water at  $4^{\circ}$ C as 1; 0.00120, the density of air at  $4^{\circ}$ C. As density d represents the weight in grams of 1 c.c. of the solution, the concentration c may be obtained by multiplying the density d by the percentage p, that is, c = p.d. By substituting p.d. for c in the formula II, we obtain the formula:

III. [a] = 
$$\frac{100 \text{ a}}{l.p.d}$$
.

Example: for cane-sugar, when  $a=34.68^{\circ}$ , l=2, p=23.68, and d=1.1,  $[a] = \frac{100 \times 34.68}{2 \times 23.68 \times 1.1} = 66.57^{\circ}$ . This formula is used

when the observation is to be made with solutions of various percentages.

The specific rotation of some substances remains constant at different strengths of the solutions. The concentration of such solutions may be found from the formula:

IV. 
$$c = \frac{100 a}{l \cdot [a]}$$
.

When the specific rotation or the value of [a], the length of column of solution l, and the observed angle a are known, the concentration c may be calculated readily. Example: for cane-

sugar, when 
$$a=34.68^{\circ}$$
,  $l=2$ , and  $[a]=66.57^{\circ}$ ,  $c=\frac{100\times34.68}{2\times66.57}=26.048$  grams.

The specific rotation of the great majority of active substances varies according to the strength of the solution at different rates for different substances. These variations are said to be due to (1) electrolytic dissociation in water solutions, (2) the formation or decomposition of molecular aggregations of simple structure, (3) the presence of complex polymerized molecules in the solution, (4) the combination of an active body with solvent,—hydrates, (5) hydrolysis, (6) small variations in the atomic equilibrium of the active molecule, and other unknown causes.

Saccharimeters.—Any of the polariscopes heretofore described may be used for the determination of the strength of sugar solutions. The concentration may be calculated from the for-

mula 
$$c = \frac{100 \text{ a}}{l.[a]}$$
 and the percentage from the formula  $p = \frac{100 \text{ a}}{l.d.[a]}$ .

It is, however, extremely desirable to be able to use ordinary white light and to read the percentage of sugar content directly on the scale. This desire led to the construction of special instruments for sugar analysis; they are called *Saccharimeters*.

Single Quartz Wedge Compensation.—The use of white light was effected in 1848 by Soleil, by inserting a quartz compensation wedge between the polarizer and the analyzer. The prin-

ciple of this contrivance is based on the dextro- or levo-rotatory power of quartz crystal when cut perpendicularly to its axis. When the dextro-rotatory substance is inserted between the polarizer and the analyzer which were set on the zero-point, the optical equilibrium or uniform shade will instantly be destroyed. If we now insert, between the polarizer and the analyzer, a levo-rotatory quartz plate of such thickness that its rotation counteracts exactly that of the dextro-rotatory substance, the optical equilibrium will be restored. It may then be said that the rotation of the dextro-rotatory substance has been compensated by that of a levo-rotatory quartz plate. Levo-rotatory substances may be compensated by a dextro-rotatory quartz plate.

The Soleil contrivance consists of two quartz plates, cut perpendicularly to the axis of the crystal, of exactly the same thick-

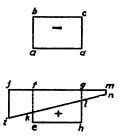


Fig. 22.—Single quartz compensation wedge.

ness and of opposite rotatory power. The dextro-rotatory plate is split into two wedges so that its thickness may be reduced or increased at will by sliding along in order to compensate within certain limits any rotation. Fig. 22 shows a plan view of the compensation device; a b c d represents the levo-rotatory plate 4.3 millimeter thick; e f g h, the dextro-rotatory plate. The two plates are equal in thickness. The dextro-plate is cut along k l, forming two wedges e k l h and g l k f with an equal wedge angle of g. The wedge g l k f is replaced by a larger wedge m n i j which has a wedge angle of equal dimension. After a polariscope has been so adjusted that the analyzer remains exactly on optical equilibrium or the zero-point, insert these plates between the polarizer and the analyzer, keeping the two wedges

of the dextro-plate in such a position that e f will remain equal to a b of the levo-plate; there will be no change in the optical equilibrium as the degree of rotation and the dispersion of rays are the same for both dextro- and levo-rotatory quartz. we slide the larger wedge m n i j to the right, the optical equilibrium will instantly be destroyed, as the dextro-plate exceeds the levo-plate in thickness and consequently in rotation. This excess of dextro-rotation may be used for compensating the levo-rotation of some active substance. If we slide the larger wedge to the left, the optical equilibrium will also be destroyed, as the levo-plate exceeds the dextro-plate in thickness and rotation. This excess of levo-rotation may be employed for compensating the dextro-rotation of some active substances. may be shown by an example; after restoring the optical equilibrium by sliding back the larger wedge, insert a cane-sugar solution between the polarizer and the analyzer; the optical equilibrium will be destroyed instantly, but it will be restored when the larger wedge is moved to the left until the sum of its rotation and that of cane-sugar will be equal to the rotation of the levo-plate. In the construction of a polariscope, these plates are placed just behind the analyzer. The levo-plate a b c dand the wedge  $e \ k \ l \ h$  are fixed; the larger wedge  $m \ n \ i \ j$  is mounted in a brass frame provided, on the lower edge, with a lateral rack in which works a pinion controlled by button, so that the wedge may be slid horizontally to the right or left. The two wedges are so arranged that the faces j m and e h are perfectly parallel to each other. The space between the two wedges is made as small as possible, for the air occupying the space would cause a lateral displacement of the ray, that is, the ray passing through the wedges will be bent by the air, owing to its density being rarer than that of quartz, as shown in Fig. 23.

It has been demonstrated that the rotation dispersion of canesugar is very nearly the same as that of quartz. This made it possible to use white light in the observation of cane-sugar in a polariscope provided with a quartz compensation wedge and a Lippich or Jellett half-shadow polarizer. In such a polariscope, not only cane-sugar, but any other substance having nearly the same rotation dispersion as that of quartz may be observed.

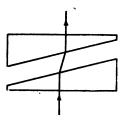


Fig. 23.—Diagram showing lateral displacement of ray.

The device of the above description is known as single wedge compensation.

Double Wedge Compensation.—Double wedge compensation has been introduced into the saccharimeter by Schmidt and Haensch. In this contrivance, as shown in Fig. 24, the levoplate of the single wedge compensation is replaced by two levowedges so that both the dextro- and levo-plates may have variable thickness. The smaller wedges are fixed while the larger wedges are arranged movable in the same manner as was the one in the single wedge compensation.

The advantage of this contrivance is that a levo-rotation of

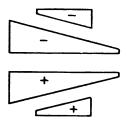


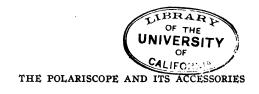
Fig. 24.—Double quartz compensation wedges.

greater range may be compensated by this combination. Another advantage is that, in this combination, the optical equilibrium is not fixed at one point; it may be set at any point, within certain limits, by moving the larger wedges in such a way that the thick-

ness of the dextro-plate becomes equal to that of the levo-plate. This makes it possible to observe the rotation of active substances at different parts of the larger wedges and diminishes errors by taking the means of several observations.

Graduation of Circular Scale for Cane-Sugar.—It is very important from the practical standpoint to graduate a scale in such a manner that the graduation shows directly the per cent of sugar contained in a substance examined.

Dissolve N grams of pure cane-sugar in a 100 c.c. flask with water and make up exactly to the 100 c. c. mark at 20° C, observe it in a 200 millimeter (m. m.) observation tube, using sodium light and designate the observed angle by b. Assume next that we have a solid or liquid substance to be investigated which contains p grams of cane-sugar in 100 grams, that is, p per cent of cane-sugar, the balance consisting of non-sugars which are optically inactive and soluble in water; weigh exactly N grams of this substance, dissolve in water, make up exactly to 100 c. c. at 20° C, observe in a 200 m. m. observation tube, and designate the observed angle by c. Since the concentration of the pure cane-sugar solution is N and that of the impure sugar solution, o.o. No, the proportion of their rotations and concentrations would be b:c::N:0.01Np, from which we obtain b:c::100:p. It is evident from this proportion that the observed angle c is directly proportional to the percentage p. If we assume b=100, c will be equal to p and indicate directly the percentage of sugar. It does not matter how many circular degrees the angle b may include, for the number 100 does not necessarily represent 100 circular degrees, but simply the angle of rotation when the substance containing 100 per cent of cane-sugar is observed under the conditions specified above; therefore, if we divide the angle b into 100 equal parts on the circular scale, each division will indicate I per cent of pure cane-sugar, and when N grams -of a substance consisting of sugar and non-sugar is made up to 100 c. c. with water and observed under the conditions above specified, the reading on the scale will indicate the per cent of cane-sugar in the substance.



Graduation of Scale for Saccharimeter with Compensation Wedges.—The principle described above may also be applied to the sugar-scale with compensation wedges. Turn the analyzer until a perfect optical equilibrium is obtained, then insert the Soleil wedge compensation contrivance and move the frame carrying the larger wedge until a perfect optical equilibrium is restored, make a mark at some convenient place on the frame carrying the larger wedge and designate this mark as the zero-point; prolong this mark over to a fixed frame work which carries no optical part; this mark will serve as the fixed index. This is shown by (a) in Fig. 25; O on the movable frame S indicates the zero-point; O on the fixed frame I, the fixed index.

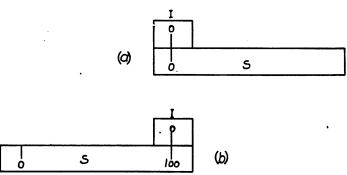


Fig. 25.—Graduation of sugar scale.

Now insert a 200 m.m. observation tube filled with a cane-sugar solution, whose strength is such that 100 c. c. contains exactly N grams of pure cane-sugar at 20° C, move the frame carrying the larger wedge until a perfect optical equilibrium is obtained, then make, on the movable frame, a mark exactly opposite the fixed index mark. This mark is designated as 100, as shown by (b) in Fig. 25. When the space between 0 and 100 is divided into exactly 100 equal parts, each division represents 1 per cent of cane-sugar. If we weigh out exactly N grams of a substance containing a certain per cent of cane-sugar, make up to 100 c. c. with water at 20° C, observe through a 200 m. m. tube, the number of divisions read on the scale will give directly the amount of sugar in per cent. This N grams of cane-sugar is

called *Normal Weight*; the solution containing N grams of cane-sugar in 100 c.c. at 20°C, *Normal Solution*. A quartz plate of such thickness that its rotation exactly corresponds to that of normal solution is called *Normal Quartz Plate*. This plate is preferred to normal sugar solution in graduating a sugar-scale; for, once made, it is always ready to be used and variation of temperature does not affect the rotation, while it requires much time to prepare normal solution and it is very difficult to keep the solution in normal condition.

The Ventzke Sugar-Scale.—There are two sugar-scales in common use; one is a French scale, the other, a German scale, known as the Ventzke scale. In the French scale, the 100 point is defined as the rotation by a quartz plate 1 m. m. in thickness. This definition, however, is unsatisfactory, as the rotation of quartz plates has been variably observed and consequently normal weight of sugar has undergone changes between the limits 16.02 and 16.47 grams.

In the Ventzke scale, the 100 point is defined as the rotation of a water solution of pure cane-sugar having a specific gravity of 1.1 at 17.5°C referred to water at 17.5°C as 1. tion of Ventzke was to render the use of a balance unnecessary in the estimation of sugar, by preparing normal solution, having a specific gravity of 1.1 at 17.5° C by means of an areometer. This. however, had not been realized, owing to the fact that non-sugars usually possess a specific gravity different from that . of cane-sugar and hence a solution of impure cane-sugar having a specific gravity of 1.1 at 17.5°C by means of an areometer. the substance that a pure cane-sugar solution having the specific gravity 1.1 does. Thus the specific gravity method for the preparation of normal solution from an impure sugar sample was found to be impracticable, but as it was not desirable to abandon the scale once introduced, the concentration of the Ventzke normal solution was determined. It was found that 100 c. c. of a water solution of pure cane-sugar having the specific gravity 1.1 at 17.5°C contained 26.048 grams of cane-sugar weighed in air with brass weights. When the same weight of pure canesugar was dissolved in water, made up to 100 c. c. at 17.5° C, and observed in a 200 m. m. tube, the 100 point of the Ventzke scale was found again. Thus the weight 26.048 grams has been decided upon as the normal weight for the Ventzke scale. The 100 point of the Ventzke scale corresponds exactly to 34.68 circular degrees for D at 17.5° C.

The graduation of the Ventzke scale and the determination of sugar in impure samples may be summarized as follows: normal sugar solution is made by dissolving 26.048 grams of pure cane-sugar, weighed in air with brass weights, in a 100 c. c. Mohr flask with water at 17.5° C; this, polarized at 17.5° C in a 200 m.m. tube, care being taken to keep the temperature of the

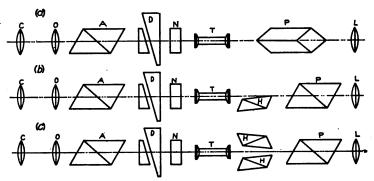


Fig. 26.—Optical parts of saccharimeters.

wedge compensation also at 17.5°C, establishes the Ventzke 100 point of the saccharimeter. When 26.048 grams of impure sugar are weighed, dissolved, and polarized under the sa:ne conditions, the Ventzke scale gives directly the percentage of pure sugar present in the substance.

Types of Saccharimeters.—There are several types of saccharimeters in use but only the half-shadow types will be presented here as they are the ones most commonly adopted in the sugar laboratories of this country. The arrangement of the essential optical parts is shown in Fig. 26; (a) represents a saccharimeter provided with a Jellett twin prism polarizer, a single wedge compensation, and a Ventzke scale; L indicates the illumination

lens; P, the Jellett twin prism polarizer; N, the levo-quartz plate; D, the dextro-wedges; A, the analyzer; O, the objective; C, the ocular; T, the observation tube. (b) shows a saccharimeter provided with a Lippich double field polarizer, a single wedge compensation, and a Ventzke scale; H indicates a half prism; (c), a saccharimeter provided with a Lippich triple field polarizer. Fig. 27 shows a saccharimeter with all the optical parts mounted in their proper places. Costly saccharimeters are provided with a Lippich double field or triple field polarizer and a double wedge compensation. All these saccharimeters are manufactured by the firm of Schmidt and Haensch of Berlin, Germany.

Principle of the Vernier and Its Use.—In order to enable the observer to accurately read the scale in tenths, sugar-scales are

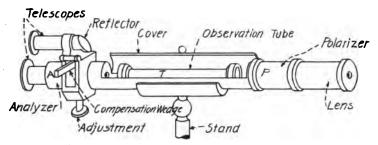
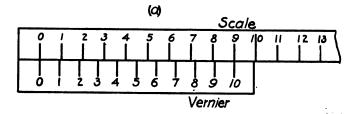


Fig. 27.—Saccharimeter with all the optical parts properly mounted.

provided with verniers. They are usually graduated on both sides of the fixed index o (Fig. 25). As shown by (a) in Fig. 28, a space covering 9 unit divisions on the scale is divided into 10 equal divisions on the vernier; therefore, each division on the vernier is equal to 9/10 of a unit division on the scale. It is evident, then, that the first division on the scale exceeds the first division on the vernier by 1/10 of a unit division, the second division on the scale exceeds the second division on the vernier by 2/10 of a unit division and so on until the ninth division on the scale exceeds the ninth on the vernier by 9/10 of a unit division. When the scale is moved to the left, the first division on the scale coincides with the first division of the vernier when the scale has been moved 1/10 of a unit division; the second division on the scale agrees with

the second on the vernier when the scale has been moved 2/10 of a unit division, and so on until the ninth division on the scale coincides with the ninth on the vernier when the scale has been moved 9/10 of a unit division. In reading the sugar-scale, suppose that the scale has been moved past 32, the fixed index remaining between 32 and 33 as shown by (b) in Fig. 28; the fraction may be read by counting the division lines on the vernier to the right until a division line which coincides with one on the scale is reached. In this case, 7 on the vernier coincides with a line on the scale, therefore, the number sought is 7 and the complete reading of the scale would be 32.7. When the fixed



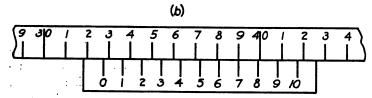


Fig. 28.—Sugar scale and vernier.

index o coincides with a unit division line on the scale, the reading will be a whole number.

Testing the Saccharimeter Scale.—The scale is a very important part of the saccharimeter as it is the chief source of error in observation. Formerly ivory scales were in common use but were abandoned, owing to the fact that their length is very easily influenced by the moisture in air, the change amounting as much as 0.3° Ventzke. Nickelin scales are now in general use as they are entirely free from the influence of moisture and also, almost independent of the change of temperature, the

change caused by 10°C of temperature fluctuation being only about 0.01° Ventzke.

It is very necessary to test all parts of the scale to determine whether or not it is correctly graduated throughout. The most suitable apparatus for this purpose is the Schmidt and Haensch control observation tube which is illustrated in Fig. 29. The tube A is made movable within the tube B, fitting accurately, and closed perfectly tight at C by the aid of a leather washer; the ends D and E are closed with a cover-glass plates and screwcaps. The tube A is moved by means of the pinion and rack F. On the rack, there is a millimeter scale which works on the

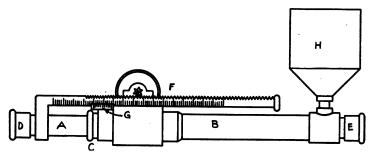


Fig. 29.-Control observation tube.

vernier G so that the tenths of a millimeter may be read off directly. The funnel H, which is detachable, serves to take up the excess of liquid expelled from the tube when it is shortened. In filling the tube, remove the funnel, close the opening with a cap, extend the tube in full, remove the cap E, pour a sugar solution in, and close E; and after the tube is brought to a horizontal position, attach the funnel H. In order to prevent evaporation of the liquid, the funnel is usually covered with a screw-cap provided with a small vent-hole.

Testing is carried on as follows: Move the compensation wedge until a perfect optical equilibrium is obtained, examine if the index o on the vernier coincides with the zero on the scale; if not, shift the index by means of an index correcting key until they coincide exactly. Now insert the control tube filled with a cane-sugar solution, extend the length of the tube

until the rotation of the sugar solution reaches  $100^{\circ}$  Ventzke exactly, call this rotation a and the length of the tube l; shorten the tube and note the length l' and the observed rotation b'. As stated previously, other things being equal, rotation is directly proportional to the length of column of sugar solution, therefore, the calculated rotation b, corresponding to the length l' would

be exactly equal to  $a \times \frac{l'}{l}$ . Should the observed rotation b' agree

with the calculated rotation b exactly, the scale is correctly graduated; should b and b' disagree, the difference must be an error of graduation and be added to or subtracted from the observed rotation, as the case requires, in order to obtain the correct value for the observation. Every point of the scale may be tested with great accuracy by this method when the temperature is kept constant.

Observation Tubes.—Fig. 30 shows a longitudinal section of an

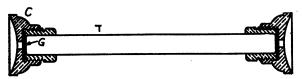


Fig. 30.—Observation tube.

ordinary observation tube. T indicates the tube made of glass or brass; G, a cover glass plate; C, a brass screw-cap. For accurate observation, a glass tube should be used, as the change in length with temperature is much less with a glass tube than with a brass tube. The standard length of an observation tube in saccharimetry is 200 m.m.; 400 m.m. and 100 m.m. tubes are also in common use; when a 400 m.m. tube is used, the reading should be divided by 2; when a 100 m. m. tube is used, the reading should be multiplied by 2. The faces of cover-glass plates should be perfectly plane and parallel. When closed with screw-caps, cover-glasses should not be pressed too hard, as the pressure will produce double refraction, which will destroy the uniformity of the field of vision and cause a displacement of the zero-point.

Pellett Continuous Flow Tube.—For such an analysis as beetjuice, wherein the sugar content does not vary in a great range, a Pellet continuous flow tube may be conveniently used. Fig. 31 shows its longitudinal section. It is usually made of brass;

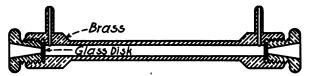


Fig. 31.—Continuous flow observation tube.

the ends are closed with glass plates and inside screw-caps. At each end of the tube is attached a small side tube which opens into the interior of the tube very close to the glass plates. To one of these side tubes is attached a small funnel and to the other a piece of rubber tubing with a properly bent glass tubing so that a solution in the observation tube may be forced out when a fresh solution is poured into the funnel. A solution may also be drawn into the observation tube by a syphon arrangement.

Observation Tube With a Water-Jacket.—When a very accurate observation is to be made, it is necessary to have a contriv-

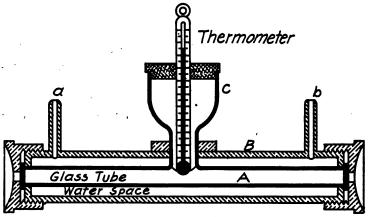


Fig. 32.—Observation tube with water-jacket.

ance to control the temperature of the solution in the observation tube during the observation. To meet this need, an observation tube provided with a water-jacket has been devised.

Fig. 32 shows a longitudinal section of the tube. The observation tube proper A is a glass tube, to the middle of which is attached, by fusing, a short thistle-tube C for receiving a thermometer. A special thermometer accurately graduated to I/IO is held in C by means of a rubber stopper. The tube C serves also as a funnel for filling the observation tube with the solution. The ends of the observation tube are closed with glass plates and screw-caps. The jacket B is made of brass. At the extremes of the jacket are attached small side tubes a and b to which small rubber tubes may be connected in such a manner that water may be circulated around the observation tube by syphon arrangement until the desired temperature of the solution in the tube is reached.

Source of Light.—Any lamp that will give a strong white light may be used for a saccharimeter such as a coal-oil lamp with duplex burner, a gas lamp or an electric lamp (32 c. p.) with a frosted globe.

## Chapter VII.

## CHEMICAL APPARATUS AND GENERAL METHODS FOR SUGAR ANALYSIS.

Chemical Apparatus.—In this work only the chemical apparatus required for the optical analysis of cane-sugar will be discussed.

Balances and Weights.—A sugar-house laboratory should be provided with two balances; one analytical balance that is sensitive to 0.05 mg. and one balance that is sensitive to 1 mg. The latter would be sufficiently accurate for the technical analysis of sugar. The balances should be adjusted from time to time, until they balance exactly by means of a screw attached to one end of the beam of the balance. The weights should be such that one set



Fig. 33.-Sugar dish.

consists of from 100 gram weight to 1 milligram weight. It is also convenient to have a set of weights especially made for sugar analysis: half-normal weight (13.024 grams), one-normal weight (26.048 grams), and double-normal weight (52.096 grams). It is also desirable to have a large platform balance with a set of weights consisting of from 1 kilogram weight to 1 gram weight for weighing a large quantity of material at a time.

Weighing Dish.—In sugar analysis, material to be investigated is conveniently weighed in a nickel capsule, commonly known as a sugar-dish (Fig. 33). These dishes are usually provided with counter-weights marked with numbers corresponding to those on the dishes. As the dishes wear, the counter-weights become too heavy and should be balanced with the dishes from

time to time, by reducing the lead particles kept in the screw handle holes of the weights.

Flasks.—There are several forms of flasks used in sugar

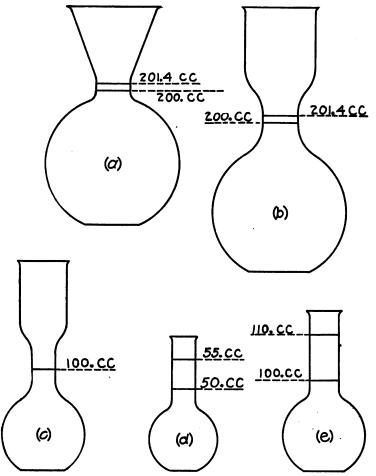


Fig. 34.—Flasks for sugar analysis.

analysis. The most convenient ones are shown in Fig. 34; (a) is Stift's flask; (b) and (c) are Kohlrausch flange necked flasks; (d) and (e) are known as sugar flasks, since they are especially made for sugar analysis. These flasks are graduated according

to Mohr cubic centimeters, in which system I c. c. is the volume occupied by I gram of pure water at 17.5°C; hence, they should be filled with a liquid at 17.5° C to the graduation mark, in order to measure the volume indicated by their denominations. In measuring a liquid, these flasks should be filled until the bottom of the meniscus comes to a level with the graduation mark as shown in Fig. 35. Not only with flasks but with all graduated apparatus, readings should be taken at the bottom of the meniscus.

Hydrometers, Baumé and Brix Spindles.—When a solid body is immersed in a liquid, the liquid always has a tendency to move the solid body upward. This upward pressure is equal to the weight of the volume of the liquid displaced by the immersed body; hence, the heavier the liquid is, the greater will be the upward pressure. The preparation of the hydrometer or the

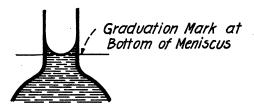


Fig. 35.-Graduation mark of flask.

areometer is based on this principle. When a spindle A (Fig. 36) is immersed in water contained in cylinder B, it will sink to the point a. If we dissolve a certain quantity of salt in this water, the spindle will rise to a certain point as b. When some more salt is added, the spindle will rise still higher. This is due to an increase in the weight and consequently in the pressure of a certain volume of the liquid displaced by the spindle. The graduation of the areometer or hydrometer of variable immersion is based on this principle. In preparing an areometer for determining the specific gravity of liquids, a point on the stem of the spindle which is exactly even with the surface of water at  $4^{\circ}$ C as a (Fig. 36) is designated as 1. After immersing the same spindle in a liquid of known specific gravity at  $4^{\circ}$  C, the point on the stem just emerging from the liquid as b (Fig. 36) is carefully marked. The interval between the two points is graduated

into a desired number of degrees. By this process, any scale for specific gravity may be prepared, using liquids whose specific gravity is accurately known. A spindle is usually made of a glass tube whose lower end is weighed with mercury. The scale is usually graduated on a piece of paper and enclosed within the stem.

A Brix Spindle is simply an areometer especially graduated for sugar solutions. In graduating the Brix spindle, the point on the stem just emerging from water at 17.5° C is designated as

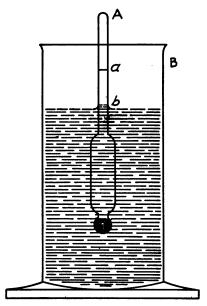


Fig. 36.—Hydrometer.

zero and the scale is so graduated that the degrees correspond directly to the percentage of cane-sugar. This is accomplished by immersing a spindle, whose zero-point is already established, in a cane-sugar solution of known percentage at 17.5° C and graduating the interval between the two points just established into a desired number of degrees. Graduation is usually carried to 1/10°. The following Brix spindles are convenient for use in sugar-house laboratories: 0-5, 0-10, 5-15, 10-20, 15-25, 20-30,

25-35, 30-50, 40-60, 50-70, and 60-90. Costly grades of Brix spindles are provided with thermometers. As the graduation of the Brix spindles is made at 17.5° C, the reading of the Brix degree of liquids should be taken at that temperature. If taken at any other temperature, the reading should be corrected for 17.5° C according to Table I, which gives the correction of Brix degrees for temperature.

Although the Brix spindle is graduated according to the percentage of cane-sugar in a water solution, it is usually used for determining the total solid matter in an impure sugar solution. Strictly speaking, the Brix degree does not indicate the total solid matter, since the specific gravity of non-sugars is different from that of cane-sugar. However, it is sufficiently accurate for technical purposes to regard the reading as approximate total solid matter. The degree observed is called Brix DEGREE or simply, "Brix." The process of determining the Brix degree by means of this spindle is called "Brixing."

A Baumé Spindle is an areometer so constructed that, when immersed in pure water at 12° C, it sinks nearly to the top of the stem. The point thus determined is the zero-point of the scale. It is then immersed in a solution of 15 parts of salt and 85 parts of water, whose density is about 1.116, and the point on the stem to which it sinks is marked 15. The interval between these two points is divided into 15 equal parts. The graduation is continued to the bottom of the stem which is about 50.

Table II, which has been prepared by Stammer, gives the comparison of these three areometer degrees at 17.5° C.

In using these areometers, the scale should be read at the bottom R but not at the top R' of meniscus (Fig. 37).

Westphal Balance.—Another apparatus employed for the determination of specific gravity is the Westphal balance, a very convenient and fairly accurate form. Its principle and construction may be considered briefly here since it is frequently used in sugar-house laboratories. Fig. 38 represents the simplest form of this balance. The column C may be extended or shortened by loosening a screw s. The beam may be leveled by means of a screw e at the base of the column. The front half

of the beam is divided into 10 equal parts. The beam carries a pointer at its back end to be used in balancing the beam by bringing it exactly opposite the index i attached to the frame. The beam also carries, at its front end, a hook upon which a glass plummet B carrying an accurate thermometer is suspended by means of a platinum wire. The plummet is so designed that it will displace about 5 grams of pure water. The balance is provided with 5 riders or weights, a, a', b, c, and d, as shown in Fig. 38. a and a' each weighs exactly the same as the volume of water displaced by the plummet. b, c, and d weigh respectively one-tenth, one-hundredth, and one-thousandth the weight of a or a'. The balance should be so adjusted, by means of the screw e, that

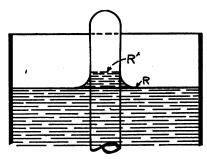


Fig. 37.—The point of reading an areometer scale.

when the plummet is immersed in pure water at 17.5°C and a or a' is suspended on the hook together with the plummet, it will be in perfect equilibrium; at this position, the weight a or a' has the specific gravity value 1.0000; when b, c, and d are suspended on the hook, their specific gravity values are respectively 0.1, 0.01, and 0.001; when a, b, c, and d are placed on the first division of the beam, their values are respectively 0.1, 0.01, 0.001, and 0.0001; when placed on other divisions, as, for example, division 4, their values are respectively 0.4, 0.04, 0.004, and 0.0004. Each rider is provided with hooks at the ends so that additional weights may be suspended thereon in case more than one rider are required for one position.

In using this balance for determination of the specific gravity of a liquid, suspend the plummet and the weight a on the hook

in such a manner that the former is completely immersed in the water contained in a cylinder, and bring the beam to an exact equilibrium by means of the screw e; then empty the water, dry the plummet and cylinder completely, introduce a liquid to be investigated into the cylinder, keeping the plummet and weight a the same as before; now place the weight a' on division a. Should it be too light, move it to division a and so on until a division is reached on which it happens to be too light but too heavy to be moved forward to the next division. Proceed with

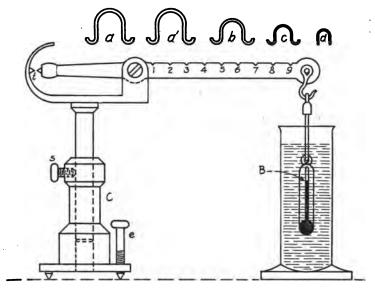


Fig. 38.—Westphal balance.

b, c, and d in the same manner until a perfect equilibrium is obtained. Should any one of these weights happen to be too heavy at division 1, set it aside and proceed with the next largest. When we find, for example, a at division 4, b at 3, c at 2, and d at 8, the specific gravity of the liquid would be 1.4328.

Pyknometer.—When a very accurate determination of specific gravity is required, a pyknometer is used. There are several forms of pyknometers. The most commonly adopted one is represented in Fig. 39. It is simply a small glass bottle of about

100 c.c. capacity. A fine thermometer T ground into the neck of the bottle serves as stopper. The bottle is provided with a side tube s with a cap c.

The method for determination of specific gravity of a sugar

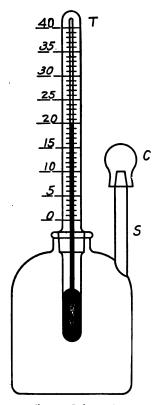


Fig. 39.—Pyknometer.

solution by means of this bottle may be briefly stated as follows: take off the thermometer and cap, clean and dry the bottle perfectly by rinsing with water, alcohol, and ether successively, and finally by blowing air into it by means of bellows; replace the thermometer and cap, weigh the whole on an accurate balance and note the weight. Now take off the thermometer and cap, fill the bottle with pure water at a temperature a little lower

than 17.5°C, replace the thermometer, and allow the bottle to stand until the temperature of the content reaches 17.5°C, place the cap on the side tube instantly, then wipe the bottle perfectly dry, weigh exactly, and note the weight. The weight just obtained minus the weight of the bottle gives the weight of water. Now determine the weight of a sugar solution to be investigated under exactly the same conditions. The weight of the solution just obtained divided by that of water will give the specific gravity of the solution.

Beakers and Funnels.—The beakers and funnels most con-

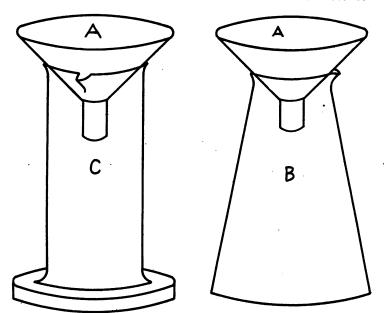


Fig. 40.—Beakers and funnels used for filtering sugar solutions.

veniently used for filtering sugar solutions are illustrated in Fig. 40. A is a glass funnel about 3 inches in diameter. It is short-stemmed or stemless. B is a thick glass beaker of 150 c.c. (½ pint) capacity. C is a short, small, glass cylinder of about the same capacity. Both forms are very commonly used in sugar-house laboratories and are sometimes called precipitating jars.

For analytical work, thin glass beakers of good quality must be used so that solutions of various kinds may be heated over a flame or on the hot water bath without breaking; their sizes are all the way from 50 c.c. to 1000 c.c. capacity.

Cylinders.—The cylinders most commonly used in sugar laboratories are of glass; their capacities vary all the way from 200 c.c. to 500 c.c. The most convenient size is about 300 c.c. capacity. Tin cylinders are also used.

Thermometer.—Either Centigrade or Fahrenheit thermometers may be employed in sugar-house laboratories, but the former are preferred to the latter as they are more convenient as well as more scientific. Two forms are in common use; one is in the form of a glass rod, the other, of a thin glass tube which floats in water.

Pipettes.—A pipette is one of the most convenient pieces of apparatus employed for measuring and delivering liquid substances. There are several forms. The most common forms are the ones graduated to measure a fixed volume of a liquid, the capacity ranging from 5 c.c. to 100 c.c. Some pipettes are so graduated that they can be used for measuring variable volumes of a liquid.

There is another form of pipette especially designed for measuring sugar solutions; it is known as a sucrose pipette and is represented in Fig. 41. It is so graduated that it will measure a double-normal weight (52.096 grams) of a sugar solution of certain percentage, if we fill the pipette with the solution to a certain mark on its stem, corresponding to the degree or percentage of total solid matter indicated by a Brix spindle without correcting for temperature. The graduation is usually made between 10 and 20 per cent in tenths. In using this pipette for measuring sugar solutions, test the solution with a Brix spindle and note the degree or percentage of total solid matter, as, for example, 15.5; now wash the pipette if necessary, rinse it out with the solution to be measured, then fill it up to the 15.5 mark by suction and transfer to a flask. A sucrose pipette

delivers a double-normal weight without being rinsed into the flask.

Burettes.—Another very convenient apparatus for measuring liquids is a burette. There are several forms. A very common one is represented in Fig. 42. Their usual capacities are

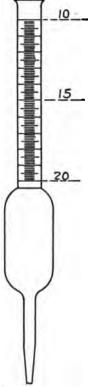


Fig. 41.—Sucrose pipette.

25 c.c., 50 c.c., and 100 c.c., graduated in tenths. An automatic filling burette may be used conveniently when rapid work is required. Fig. 43 represents an automatic filling burette fitted with a Wolf bottle for use; if the rubber bulb be pressed gently and then released when a liquid slightly exceeds the zero mark, the burette will be filled to the zero mark. This burette is very suitable for titrating the alkalinity of the juice.

Grinders.—There are several forms of grinders suitable for grinding various kinds of beet samples. A horse-radish grater is very suitable for grinding whole beets. Should the sample need to be ground fine, some good fine rasp should be used. Keil's cylindro-divider is very suitable for cossettes or other frag-

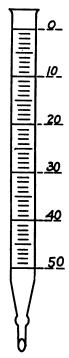


Fig. 42.—Burette.

ments of beets; Pellet and Lomont's conical rasp, for whole beets; Gallois's La sans pareille is also very well recommended for grinding cossettes. A sausage-grinder grinds cossettes and pulp sufficiently fine for ordinary purposes.

Boring Rasp.—When it is desired to take a small portion of a sample from a beet, as in the case of analysis of seed-beets, Keil and Dolle's boring-rasp may be used. Fig. 44 represents the essential portion of the boring-rasp. The borer carries at one end a hollow and detachable conical rasp provided with a

narrow opening for receiving the sample. The other end is attached to a rapidly revolving machine. In taking a sample, one should press a beet against the rasp, holding the former in such a manner that the rasp bores in diagonally as shown in Fig. 45. It is very important to bore diagonally in such a way as to include several zones, for the sugar content varies at

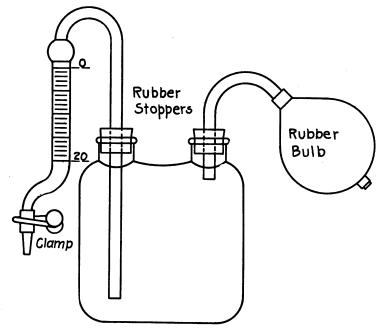


Fig. 43.—Automatic filling burette for juice alkalinity test.

different zones and lateral boring would not give a representative sample of the whole beet. When drilled through, the cone is detached and the sample is transferred to a suitable dish; then the cone is cleaned and placed back for another boring.

General Methods for Optical Analysis of Cane-Sugar.—There are numerous samples to be examined for sugar in the sugar-house and each requires particular treatment before observation. Many points, however, are similar in all polarizing operations and will be described in this article. Three methods are more or less

generally applied in the optical analysis of cane-sugar:—Gravimetric method, sucrose pipette method, and volume method.

Gravimetric Method.—In the optical examination of a substance for cane-sugar, normal solution or a solution containing one normal weight (26.048 grams) of that substance in 100 c.c. must be prepared. In the gravimetric method, normal solution is obtained by weighing out 26.048 grams of the sample, dissolving

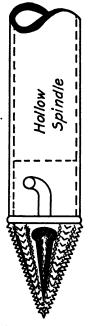


Fig. 44.—Boring rasp.

it in water, and making it up to 100 c.c. It makes no difference whether the substance to be examined is solid or liquid.

Weighing.—Adjust the balance to be used until it attains a perfect equilibrium by means of a screw attached to one end of the balance beam, place a sugar-dish in one of the weighing pans and a counter-weight with the corresponding number in the other pan and see that they balance exactly; now place a normal weight or 26.048 gram weight in the pan in which the counter-weight is

placed and transfer the substance to be examined to the sugardish by means of a glass tubing, if liquid, or a spatula, if solid, until the balance is in perfect equilibrium. When a very dark substance is to be examined, one-half normal weight (13.024 grams) should be taken. In case a substance very low in sugar content is to be examined, double normal weight (52.096 grams) should be used.

Dissolving and Transferring.—To the substance weighed out in the manner cited above, add a small amount of water, mix

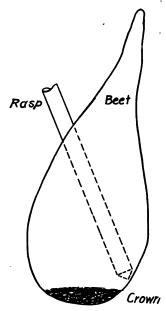


Fig. 45.—The manner of boring out sample from a beet.

thoroughly by means of a glass rod, and transfer to a 100 c.c. flask completely, by rinsing the sugar-dish with water. Should the substance be raw sugar or fillmass, every crystal should be dissolved before it is transferred to the flask. The volume of the solution should not exceed 75 c.c. when the transferring is completed.

Necessity of Clarification.—As was stated under Constituents of Non-Sugars, beet-juice contains many compounds other than cane-

sugar, and also all sugar-house products, excepting white sugar, contain these non-sugars in greater or less quantities. Some of these non-sugars are optically active and interfere with the optical analysis of cane-sugar. Coloring matter is also very objectionable, since it is impossible to make an observation when it is present in large quantities. It is, therefore, very important to remove these non-sugars as much as possible with a suitable reagent. It has been found that subacetate of lead is a very suitable reagent for this purpose since it precipitates many of these non-sugars as well as coloring matter.

Preparation of Lead Subacetate Solutions.—There are lead subacetate solutions of various strengths in use in sugar-house laboratories. The solution most commonly used may be prepared as follows: weigh out 300 grams lead acetate and 100 grams litharge, dissolve the whole in 1000 c.c. water, stir, and heat gently until all is dissolved, and call this solution "dilute lead subacetate solution." When it is cool, transfer the solution to a glass bottle provided with a syphon for drawing the solution. When a larger quantity of lead subacetate solution is required, as is usually the case, lead acetate, litharge, and water should be so proportioned as to produce a solution of the above strength. Lead subacetate solution of this strength, however, is not strong enough for those samples with much non-sugars and coloring matter, such as beet-juice and molasses. For such substances, lead subacetate solution of double strength is prepared, by dissolving 600 grams lead acetate and 200 grams litharge in 1000 c.c. water. This solution is called "concentrated lead subacetate solution." These solutions are called "lead" for simplicity.

The Amount of Lead Subacetate Solution to be Added For Clarification.—It is difficult to specify the amount of lead subacetate solution to be added for clarification as the substances to be examined vary in color and in the amount of non-sugars. Should the substance be light colored, from 5 to 10 c.c. of dilute lead solution would be sufficient. Should the substance be very dark, from 5 to 10 c.c. of concentrated lead solution should be

added. By a little experience, one may soon learn about how much lead solution to add to a certain quantity of a substance. To 100 c.c. of beet-juice or one normal weight of molasses, 10 c.c. of concentrated lead solution should be added.

Influence of Lead on the Optical Activity of Cane-Sugar.—The amount of subacetate of lead has practically no influence on the optical activity of cane-sugar in water solution under ordinary circumstances. A very large quantity of lead subacetate slightly diminishes the rotation, probably due to the formation of sucrate of lead. In alcoholic solution, however, there is a decided diminution of the dextro-rotation of sugar in the presence of any lead salt. This may be explained by the fact that insoluble sucrate of lead is readily formed in alcoholic solution.

When a substance under investigation contains a considerable quantity of invert sugar, a large quantity of lead subacetate increases the dextro-rotation of the sugar solution. This is due to the separation of levulose from invert sugar, forming insoluble levulosate of lead and liberating dextrose which will help to increase dextro-rotation.

Clarification, Completion of Volume, and Filtration.—Having transferred the substance to a 100 c.c. flask completely, add lead solution (as many c.c. as may be necessary for clarification) a little at a time and shaking thoroughly after each addition, fill with water up to the shoulder of the flask, bring the temperature of the solution exactly to 17.5° C, add a few drops of ether in order to destroy the foam, and then complete the volume to the 100 c.c. mark with water; now close the flask, mix the content thoroughly by shaking and filter, using suitable filter-paper, beaker, and funnel. The first part of the filtrate should be rejected or poured back on the filter as it is usually slightly cloudy.

Errors due to the Volume of Lead Precipitate.—The actual volume of the normal solution, obtained in the manner cited above, is a little short of 100 c.c. as the precipitate of lead occupies some space in the flask. This will naturally tend to give a higher reading than true normal solution. The error thus introduced, however, is offset, to great extent, by compensating errors,

especially in the analysis of low products. According to Sachs, acetates of potassium and sodium, formed by acetic acid which was set free in consequence of the decomposition of lead subacetate, have a tendency to diminish the dextro-rotatory power of cane-sugar. The error due to the volume of lead precipitate is disregarded in all practical work as it is very slight. When very accurate work is required, it is advisable to follow Bigclow's method. In this method, a necessary quantity of dry subacetate of lead is added to the substance after it is made up to normal solution with water. The method for determining the volume of lead precipitate will be discussed in the chapter on Special Analysis.

Observation.—Having obtained a clear filtrate, clean an observation tube, wipe cover glass plates perfectly dry and clean, close one end of the tube with one of the cover glass plates and a screwcap, rinse the tube with the filtrate twice, fill it heaping full, close with a cover glass plate and screw-cap, care being taken that no air bubble remains in the tube; wipe the tube first with a wet cloth and then with a dry towel so that the polariscope may not be soiled.

The observation is best made in a dark room. The lamp for the polariscope should be kept outside the room and light be allowed to come in through a small hole. The polariscope should be kept at such distance from the lamp that its illumination lens remains about 6 inches away from the source of light. ocular sharply, slide the compensation wedge until the field of vision is uniformly shaded. See if the zero mark comes exactly even with the index; if not, adjust the index by means of the index correcting key until it coincides with the zero mark exactly. Now place the observation tube in the polariscope, move the compensation wedge until a perfectly uniformly shaded field is obtained, and read the scale according to the direction given under PRINCIPLE OF THE VERNIER. The reading on the scale will give directly the percentage of sugar contained in the substance contained. Hereafter the word "Polarize" will be used, for the sake of simplicity, for the process of filling an observation tube and observing the rotation in a polariscope.

Sucrose Pipette Method.—As stated previously, a sucrose pipette delivers double-normal weight (52.006 grams) when filled to the mark so graduated as to correspond to the Brix degree of a solution to be examined. The process of making normal or double-normal solution by means of a sucrose pipette is very simple and may be applied to any sugar solutions whose percentage of total solid matter of Brix degree ranges between 10 and 20 per cent. The method is especially adapted for the estimation of sugar in beet-juice. The manipulation may be conducted as follows: determine the total solid matter or Brix degree of the solution to be examined by means of a Brix spindle and without correcting the Brix degree for temperature, fill the pipette with the solution by suction to the mark corresponding to the Brix degree just noted. When the sucrose pipette is wet, it should be dried or rinsed out with the solution to be measured before filling. Then transfer the solution to a 100 c.c. flask, disregard rinsing the pipette into the flask, add from 5 to 10 c.c. dilute lead solution, according to the color of the solution, complete the volume with water to the 100 c.c. mark at 17.5° C, using a few drops of ether if necessary, mix thoroughly by shaking, filter, and polarize. The polariscope reading divided by 2 will give the percentage of sugar in the solution. Should the volume of the solution be made up to 200 c.c., as is often done, the polariscope reading will give the percentage of sugar directly.

Volume Method.—So far as manipulation is concerned, this is the simplest of all the methods used in the estimation of sugar in a solution and can be applied to any sugar solution having Brix degree less than 30.0. In this method, a polariscope reading for 100 c.c. of a solution to be investigated is first obtained and this reading is reduced by calculation to that of normal weight or 26.048 grams of the solution which gives the sugar content in per cent. The process and principle may be stated as follows: determine, by means of a pyknometer or Westphal balance, the specific gravity of a solution to be investigated at 17.5°C; or take the Brix degree with a Brix spindle at 17.5°C; correct, if taken at any other temperature, according

to Table I, find the specific gravity corresponding to the observed Brix degree in Table II, and note it down. Now fill a 100-110 c.c. flask with the solution to the 100 c.c. mark, make it up to exactly 110 c.c. with lead subacetate solution, mix thoroughly by shaking, filter, polarize, and note down the reading. we have diluted the solution with 10 c.c. lead subacetate solution, the polariscope reading for the undiluted solution may be obtained by adding, to the reading just obtained, its one-tenth. If we multiply the sum by 0.26048, the product will be the polariscope reading for 26.048 c.c. of the undiluted solution. the specific gravity of sugar solution under investigation be the same as that of water, which is 1, 26.048 c.c. of the solution would weigh exactly 26.048 grams, but being, in fact, higher than that of water, 26.048 c.c. of the solution would weigh  $26.048 \times d$  grams when d designates its specific gravity. The number of c.c. of the solution, which weighs 26.048 grams and which may be designated by N, can be obtained from the proportion: 26.048 c.c. : 26.048  $\times$  d grams :: N : 26.048 grams, hence,

$$N = \frac{26.048 \text{ c.c.} \times 26.048 \text{ grams}}{26.048 \times d \text{ grams}} = \frac{26.048 \text{ c.c.}}{d}.$$

It is evident, then, that the number of c.c. of the solution weighing 26.048 grams is obtained by dividing 26.048 c.c. by the specific gravity of the solution and consequently the polariscope reading for normal weight (26.048 grams) may be obtained by dividing the polariscope reading for 26.048 c.c. by the specific gravity of the solution. This process of calculation may be represented by the following formula:

$$\frac{\left(P + \frac{P}{10}\right) \times 0.26048}{d} = S,$$

in which P designates the polariscope reading and S, the per cent of sugar in the solution investigated. This may be illustrated in numerical values: assuming that P=58.6, Brix degree=18.2, d=1.0753, sugar content would be

$$\frac{(58.6 + 5.86) \times 0.26048}{1.0753} = 15.59 \text{ per cent.}$$

A table (Table III) has been prepared from this formula by Schmidt in order to avoid this somewhat tedious calculation. The use of this table may be illustrated as follows: in the column under 18.0, the Brix degree nearest to the degree observed and opposite 58, the integral part of the polariscope reading, note the number 15.43; in the small table, opposite 0.6, the decimal part of the polariscope reading, note the number 0.16. The sum of these two numbers, that is, 15.43 + 0.16 = 15.59 would be the per cent sucrose in the solution examined.

Purity Coefficient.—As was stated under Composition of Beets, the purity coefficient is obtained by dividing the per cent of sucrose by that of the total solid matter in the juice, syrup, or other substances containing sucrose, and multiplying the quotient by 100. Since a Brix spindle is graduated according to the per cent of sugar in the solution and the specific gravity of nonsugar is different from that of sugar, the degree indicated by the Brix spindle would not represent the true total solid matter; but, in common practice, it is taken for approximate total solid matter in order to simplify the process. The purity coefficient is obtained by dividing the per cent of sugar by the Brix degree corrected for temperature and multiplying the quotient by 100.

This may be duly represented by the formula,  $P = \frac{S \times 100}{B}$ , in

which P represents the purity coefficient, B, the Brix degree, and S, the per cent of sugar. Taking 18.2 for B, 15.59 for S, P

would be  $\frac{15.59 \times 100}{18.2}$  = 85.66. The purity thus obtained is

called Apparent Purity, in order to distinguish it from Irue Purity, which is obtained by determining the actual dry substance by weighing a sample and drying it in an oven. The method for the determination of the actual dry substance and the true purity will be described in the chapter on Special Analysis. The author has prepared a table (Table IV) in which the per cent of sugar and the apparent purity may be found simultaneously. The use of this table may be shown by an

example, using the numerical values 18.2 for the Brix degree and 58.6 for the polariscope reading. For the per cent of sugar, take the number 15.6 in the column under Sugar in Juice and opposite the Polariscope Reading 58.6. For the purity, take the number 85.6 in the column under the Brix Degree 18.2 and opposite the Polariscope Reading 58.6. When a number exactly corresponding to a polariscope reading is not found in the table, the nearest number should be taken. This table may be used for any solution whose Brix degree ranges between 7 and 28 and whose purity, between 49 and 96 per cent.

## Chapter VIII.

## PRACTICAL OPERATION OF A BEET-SUGAR HOUSE.

This chapter will be devoted to the discussion of the methods of chemical control and the practical operation of sugar-house stations.

Time for Harvesting Beets.—The first thing required of the sugar-house chemist is the analysis of beets from the field in order to watch their growth and determine the suitable time for harvesting. The time for harvesting varies according to local conditions; for example, on the Pacific coast, it begins as early as the middle of July, while beets could not be harvested till the first part of September in the middle West. The ripening of beets could not be determined by the sugar content alone, as the sugar content varies according to the conditions of soil and weather, even in the same locality. It may usually be judged from their appearance. When they ripen, their lower leaves turn brown and wilt; the upper leaves turn decidedly yellower, cease to grow, and tend to lay flat on the ground; the center leaves remain stunted. Although chemical analysis alone does not determine the time for harvest, it is very important to keep the sugar-house management informed of the sugar content in beets and the quality or purity of the juice by chemical analysis. Another important matter to be taken into consideration in the management of a sugar-house is the quantity of beets available. When a long campaign is expected on account of the large tonnage of beets to be worked, the operation should begin as early as the beets could be profitably worked, for, as the season grows late, beets freeze, ferment, and not only lessen the sugar yield, but offer various difficulties to the process of manufacturing.

Sampling Beets from the Field.—The sugar content in beets varies according to the size and shape of individual beets, the



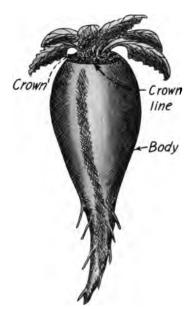


Fig. 47.—Different parts of beet.

condition of the soil surrounding them, diseases, injuries, and so forth, even in the same field; hence, it is extremely important to take a sample from a field in such a manner that the analytical result of the sample would nearly represent the sugar content in the beets of the entire field. The following method is in common practice: choose a certain number, count, beginning with the first beet of the first row, and pull the beet which corresponds to the number chosen; cross a certain chosen number of rows, count again, beginning with the beet opposite the one just taken and pull out the beet corresponding to the chosen number. Continue in this manner until a sample of 10 beets is obtained. Num-

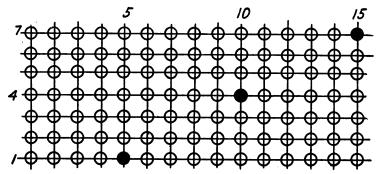


Fig. 46.—Diagram showing the manner of taking sample beets from the field.

bers should be so chosen that the sampling would cover nearly the entire field. The accompanying diagram (Fig. 46) shows a case where every fifth beet in every fourth row is taken for sample.

Preparing Sample Beets for Analysis.—As indicated in Fig. 47, the uppermost portion of a beet, which is rough and green colored, is called the Crown; the line more or less distinctly separating the crown from the other portion of the beet is called the Crown Line; the portion below the crown line is called the Body. The sugar content and the purity of the juice in the crowns are generally very low and sugar, therefore, could not be profitably manufactured therefrom. It is customary to reject the crowns before beets enter the process of sugar manufacturing; hence, it is proper to detach the crowns from sample

beets before analysis. After the crowns are detached, beets are cleaned thoroughly with some stiff brush and weighed; the total weight is divided by the number of beets and the result is recorded as the average weight of individual beets. The next step in preparing a sample for analysis is grinding; an ordinary horseradish grater answers this purpose very well. The whole sample should be ground when it consists of three or four small beets. When beets are large or the sample consists of many beets, each

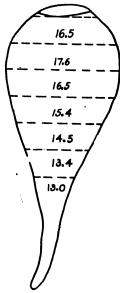


Fig. 48.—Sugar content at different zones of beet.

beet should be cut into two halves longitudinally and one-half of each beet should be taken for analysis. When the sample is still too large, each beet should be quartered longitudinally and just one-quarter of each beet be taken for analysis. The reason for cutting each beet longitudinally is to obtain the average sugar content for the whole beet. As may be seen from the illustration shown in Fig. 48, the sugar content varies at different zones of the body of a beet. It is evident then that a longitudinal section, whether one-half or one-quarter, nearly represents the average sugar content for the entire body of a beet.

Estimation of Sugar.—There are two classes of methods of analysis for the estimation of sugar in beets; namely, direct analysis and indirect analysis. In direct analysis, sugar is estimated directly in ground beets by weighing out the normal weight and polarizing it after proper treatment. This method will be described under Cossette Analysis. In indirect analysis, the juice is expressed from ground beets and the sugar is determined in the juice; the result multiplied by the juice-factor 0.95 will give the sugar content in the beets. The indirect method is generally used for the estimation of sugar in beets from fields. The sucrose pipette or volume method may be applied for the estimation of sugar in beet-juice; the volume method is preferred to the sucrose pipette method as it is the simpler and more accurate of the two.

1

After the sample is ground fine, it is wrapped in some thick, strong cloth, the juice is expressed by means of a powerful screwor lever-press and collected in a suitable vessel. It is said that the density of the beet-juice varies at different pressure; hence, the ground sample should be pressed as dry as possible in order to obtain the juice of average density. The juice thus obtained is transferred to a glass or tin cylinder and allowed to stand until all the air bubbles are expelled therefrom. Then determine the Brix degree, correct it for temperature, determine the polariscope reading, using the volume method, and find the sugar in the juice and the purity in Table IV; multiply the sugar in the juice by 0.95 and record the result as the sugar in the beets. The following data should be recorded in this work: 1, the average weight of individual beets; 2, the Brix degree; 3, the sugar in the juice; 4, the sugar in the beets; 5, the purity coefficient of the juice.

Harvesting, Transportation, Ensilage, Sampling, Taring, and Analysis.—When suitable for harvesting, beets are loosened from the ground by means of a puller, picked by hand, detached from the crowns by means of a knife, loaded in wagons, and brought to the factory or transferred to railroad cars, if to be transported to a distance. When there are more beets than could be enter-

ed into manufacture in the immediate future, they should be kept in silos, that is, they should be piled in the field and covered with earth in order to protect them from the effect of the weather until the proper time for transportation comes. When the field is situated in the immediate vicinity of the factory, the beets are brought in on wagons. One sample consisting of about 10 beets should be taken from each wagon, picking beets at random from different parts of the wagon. When beets are brought in on railroad cars, 3 samples, each consisting of about 10 beets, should be taken from each car, picking beets at random from different parts of the car. The sample beets, just as taken from wagons and cars, are usually covered with dirt and their crowns are not properly trimmed. In order to obtain the net weight of beets, weigh each sample just as it was brought in, clean the beets by washing or brushing, trim the crowns properly, and weigh them again; the loss in weight is calculated to percentage and recorded as tare. The samples thus obtained are ground and analyzed by the 110 c.c. volume method. The tare, Brix degree, sugar in juice, sugar in beets, and purity of juice should be recorded in this work.

Beet-Shed and Transportation of Beets from Beet-Shed to Factory.—The beets are unloaded in the beet-shed by throwing them with forks from the wagons or cars into the bins with sloping walls, at the bottom of which are situated flumes or sluices provided with board covers. The flumes are led, with a certain fall, to the first floor of the factory. In transporting beets, water is turned on into the flume, the board covers are removed, the beets are pushed into the current of water which will float and carry them into the factory.

Beet-Scroll, Washer, and Elevator.—The beets are carried up from the first floor into a beet washing machine by means of a spiral lift called a Beet-Scroll. The beet-washer is simply a rectangular tank provided with paddles attached to a shaft which agitate and forward the beets from one end of the washer to the other. On reaching the further end of the washer, the beets are lifted by means of a wider paddle and thrown into buckets

attached to a long endless chain called a Beet-Elevator, which carries the beets up to the uppermost floor of the factory-building.

Automatic Scale.—On arriving at the top of the elevator, the buckets empty the beets into a hopper which is so arranged as to direct the beets into an automatic scale. The automatic scale is usually so arranged as to weigh 1000 pounds in one weighing. When filled with the required quantity of beets, the scale automatically closes the bottom of the hopper and empties beets into another hopper which directs the beets into the beet slicing machine.

Beet-Slicer.—A Beet-Slicer consists of a cylinder for holding the beets and a circular disk provided with about 20 rectangular openings toward the rim for receiving knife-holders. Knife-

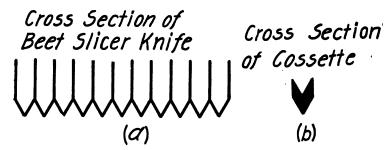


Fig. 49.—Cross sections of beet slicer knife and cossette.

holders are rectangular frames and exactly fit in the openings. The knives are fixed on the holders by means of bolts. The knives, in common use, have blades of such form as is shown in Fig. 49, (a), and cut beets in such a shape that the cross section of a slice would appear somewhat like Fig. 49, (b). The beets are piled in the cylinder, and pressed against the knives by their own weight. As the disk revolves, the beets are sliced, passed under the disk, received in a round box, thence pushed out by means of an ejector through a spout. The sliced beets thus thrown out of the slicer are received on an endless belt called a Cossette Conveyor and carried to the diffusion battery. Sliced beets are called Cossettes.

Analysis of Cossettes.—It is very important to keep the sugar-house management informed of the amount of sugar in beets entering the process of manufacture in order to compare it with the amount of sugar actually extracted from the beets. It is impossible to obtain the exact sugar content in beets by analyzing a few small samples when several hundred tons of beets varying in sugar content between wide ranges are entering the process daily. It is, however, possible that the approximate sugar content may be arrived at by analyzing the beets as frequently as permissible and taking an average result of all the analyses.

Sampling Cossettes.—The manner of taking a cossette sample is extremely important in the analysis of cossettes, for the sugar content estimated in a small sample should represent approximately that of the entire lot of beets that have entered the process during a specified interval of time. The following method is in common practice: take one hand full of cossettes while each cell of the diffusion battery is being filled, preserve it in a pail provided with a cover in order to prevent it from drying, and continue collecting the sample in this manner for two hours, or any length of time as may be specified. At the end of a period specified, take the sample into the chemical laboratory, mix the whole sample, and take a small portion for analysis.

Direct Analysis of Beets.—As stated previously, the content of marc in beets varies from 3 to 7 per cent and hence the juice-factor 0.95 is not applicable in all cases. It was also stated that the density of beet-juice varies according to the pressure applied to express the juice from ground beets and to the degree of fineness of ground beets as well. These sources of errors render the sugar content in beets estimated by the indirect method very unreliable. The only reliable method for the estimation of sugar in beets is the one in which the sugar may be determined directly in a weighed quantity of a finely ground sample of beets. The determination of the sugar content in a weighed quantity of beets is called Direct Analysis.

Direct Analysis.—There are several methods for direct analysis of beets. Those, in common use, are, I, Pellet's instantaneous

aqueous diffusion method; 2. Sachs-Le Docte's method; 3, Wojcicki's method; 4 Pellet's aqueous hot digestion method; 5, Stammer's cold alcoholic digestion method; 6, Rapp-Degener's hot alcoholic digestion method; 7, Scheibler's alcohol extraction method with Soxhlet extraction apparatus.

Pellet's Instantaneous Aqueous Diffusion Method.—For this method, the cossettes or beets should be reduced to a perfect pulp (brei) by means of a cylindro-divider, Pellet's rasp, or Gallois's La sans pareille. Weigh out exactly one normal weight of the sample, transfer to a Kohlrausch or Stift flask with a 200.6 c.c. mark, add 5 c.c. of dilute lead subacetate solution and water up to the shoulder of the flask, mix the content thoroughly by rotating the flask, allow it to stand for 25 minutes in order to expel air-bubbles, add a few drops of ether to destroy the foam on the surface of the solution, make up to the 200.6 c.c. mark with water, close the flask with the hand, shake thoroughly, filter, and polarize with a 200 m.m. observation tube. The polariscope reading multiplied by 2 will give the per cent of sugar in the beets. Some authorities recommend an acidulation of the solution with a few drops of acetic acid just before completing the volume. It has been shown that lead subacetate separate levulose from invert sugar, forming insoluble levulosate of lead; this gives undue prominence to dextro-rotation but it may be restored to normal rotatory power by acidulating with acetic acid. It has also been shown that asparagine cannot be precipitated but is rendered strongly dextro-rotatory by lead subacetate; this activity, however, may be rendered inactive by acidifying with acetic acid. The disadvantage of this method is that air-bubbles which remain in the flask adhering to the pulp tend to render the per cent of sugar unduly high. In order to minimize this error, the flask should be connected by means of a rubber stopper and glass tubing with a vacuum pump and as much air-bubbles as possible should be expelled from the liquid. Stift recommends the following method for removing air-bubbles: transfer the weighed sample to a 201.2 c.c. flask, add a lead subacetate solution, fill the flask with water about two-thirds full, shake thoroughly; then hold the flask in the left hand in an oblique position, tap it lightly with the right hand finger while continually rotating. When sufficient foam is collected on the surface of the liquid, add a few c.c. of absolute alcohol in order to disperse the foam and complete the volume to the mark, after adding a few drops of ether in order to clear the graduation mark from foam.

Sachs-Le Docte's Method.—This is a modification of Pellet's instantaneous aqueous diffusion method. In this method, a requisite quantity of water is added to the normal weight of a beet sample in a capsule in order to make normal solution instead of making it up to a required volume in a flask. It is assumed that the beets contain 95 per cent of juice with the specific gravity 1.07; the volume of the juice contained in one nor-

mal weight of beets would then be  $\frac{26.048\times0.95}{1.07}$  = 23.12 c.c.

It follows from this that the volume of water to be added to one normal weight of beets in order to make normal solution would be 100—23.12=76.88 c.c. In order to make a solution containing one normal weight in 200 c.c., as in the case of Pellet's instantaneous aqueous diffusion method, 200—23.12=176.88 c.c. of water should be added to one normal weight.

For this method, the beet should be ground as fine as for Pellet's instantaneous aqueous diffusion method. Weigh out exactly one normal weight of the sample in a copper capsule, add 5 c.c. of dilute lead solution and 172 c.c. of water, agitate vigorously so that sugar may be uniformly distributed, place a discover on the capsule in order to prevent it from evaporation, allow it to stand for three minutes, filter, and polarize in a 200 m.m. observation tube. The polariscope reading multiplied by 2 will give the per cent of sugar in the beets. This method is not theoretically correct but sufficiently close for technical purposes.

A special pipette has been devised for measuring lead solution and water in order to facilitate the operation. Fig. 50 shows the general arrangement of the apparatus. It may be manipulated

as follows: give a quarter turn to the stop-cock K to admit the lead solution through the tube B from a reservoir; when the solution reaches the 5 c.c. mark, give another quarter turn to the

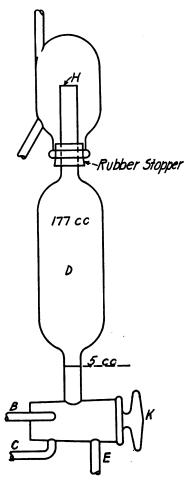


Fig. 50.—Sachs-Le Docte pipette.

cock to admit water through the tube C from a water reservoir; when the solution overflows at H, give another quarter turn to the cock. The content of the pipette thus obtained measures ex-

actly 177 c.c. and another quarter turn of the cock will discharge the solution into the capsule.

Wojcicki's Method.—The method of Wojcicki is based on the same principle as that of Sachs-Le Docte. In this method, however, one normal weight is made up to 100 c.c. with 5 c.c. of lead solution and 71.8 c.c. of water. A special pipette (Fig. 51) has also been prepared for this method. In using this pipette, 5 c.c. of lead solution is first introduced and then water is run up to the 76.8 c.c. mark. The operation of this method is car-

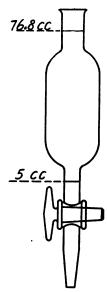


Fig. 51.-Wojcicki pipette.

ried out in exactly the same manner as that of the Sachs-Le Docte method. When the solution is polarized in a 200 m.m. tube, the polariscope reading gives directly the per cent of sugar in beets.

The methods of Sachs-Le Docte and Wojcicki, owing to their rapidity, are usually used in testing seed- or mother-beets.

Pellet's Hot Water Digestion Method.—For this method, the beets need not be ground so fine as for the instantaneous aqueous diffusion method. Cossettes may be ground with a sausage-

grinder, whole beets, with a horse-radish grater. ground beets are thoroughly mixed, weigh out double normal weight (52.096 grams) in a sugar dish, transfer to a Kohlrausch flask with a 201.2 c.c. mark by the aid of water and a glass rod, add 10 c.c. of dilute lead solution and water up to the shoulder of the flask, place it in a hot water bath, allow it to remain therein for 30 minutes, shaking occasionally and keeping the temperature at about 80°C. At the end of the time specified, take out the flask, cool the content to 17.5°C in cold water, add a few drops of acetic acid, complete the volume of the solution with water exactly to the 201.2 c.c. mark after clearing the graduation mark with ether, mix, filter, and polarize in a 200 m.m. tube. polariscope reading will give the per cent of sugar in the beets directly. The error due to air-bubbles is entirely eliminated in this method. This method, being very simple, inexpensive, and close enough to compare very favorably with other accurate methods, such as alcoholic methods, is used in sugar-house control work by most sugar-house chemists.

Alcoholic Methods.—All water methods are subject to one important error which is derived from the optical activity of some of the non-sugars present in beets such as invert sugar, raffinose, pectine substances, malic acid, tartaric acid, asparagine, glutamine, and so forth. In order to eliminate this error, alcoholic methods have been proposed. It is claimed that, in the presence of strong alcohol, a part of these compounds form insoluble salts with lead or partly lose their optical activity. The quantity of lead subacetate to be added, however, should be as small as permissible, for a slight excess of lead subacetate is sufficient to make those optically active substances strongly dextro-rotatory. Another objection to an excess of lead subacetate is that it forms inactive tribasic lead saccharate with sugar in the presence of alcohol. It should, therefore, never be used more than specified.

Stammer's Cold Alcoholic Digestion Method.—This method is very similar to Pellet's instantaneous aqueous diffusion method in the details of manipulation; the only difference is that this

method employs 92 per cent alcohol in place of water. The sample should be ground for this method as fine as for the instantaneous aqueous diffusion method. Weigh out double normal weight of the sample, transfer to a 201.2 c.c. flask with 92 per cent alcohol, add 6 c.c. of dilute lead solution, complete the volume of the solution with alcohol of the same strength, shake vigorously, allow it to stand for from 15 to 20 minutes, filter, and polarize in a 200 m.m. tube. The polariscope reading gives the per cent of sugar in the beets.

Rapp-Degener's Hot Alcoholic Digestion Method.—This method is exactly the same as Stammer's method excepting that, in this method, 4 c.c. of dilute lead solution is used instead of 6 c.c. and that the alcoholic solution, after the flask is filled about four-fifths full, is digested in a hot water bath at slow boiling for 20 minutes. The flask is usually connected, by means of a cork-stopper, with a return condenser or a glass tubing about 1 meter in length and 10 m.m. in diameter in order to return the vaporizing alcohol. At the end of the time specified, cool the flask and the contents to 17.5°C, complete the volume to the 201.2 c.c. mark, mix, filter, and polarize.

Scheibler's Alcoholic Extraction Method with Soxhlet Extraction Apparatus.—This method is the most accurate hitherto known, is regarded as a standard, and is used in scientific investigations. Most objectionable errors, such as were cited above, are eliminated in this method, for there are no air-bubbles or marc to contend with, practically no volume would be occupied by a precipitate of lead, most optically active substances are left behind, since they are mostly insoluble in alcohol and what little may have dissolved may be removed with a few drops of lead subacetate solution.

Fig. 52 represents the general appearance of the Soxhlet extraction apparatus. E represents the extraction tube; b, the vapor conducting tube; s, the syphon tube; t, the stopper for an opening at the top of the syphon tube; A, a return condenser provided with two side tubes for the inlet and the outlet of water into the water-jacket; F, a 100 c.c. flask. When the flask is filled

with alcohol and heated and water passed into the water-jacket

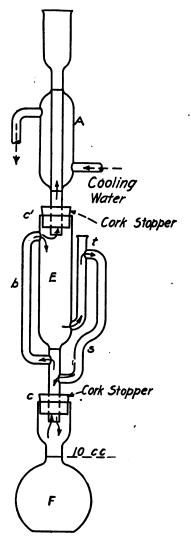


Fig. 52.—Soxhlet extraction apparatus.

of the condenser, the alcohol will vaporize from the flask, pass through the tube b and up to the condenser A, where it is con-

densed and returned to the extractor E. When the alcohol, accumulating in the extractor, reaches to the top of the syphon tube s, it will be drawn down into the flask by syphon action. In analyzing beets by this method, proceed as follows: place a plug of absorbent cotton at the bottom of the extractor E so that beet chips will not be drawn into the syphon tube, introduce completely 26.048 grams of finely ground beets into the extractor by washing in with alcohol any particles of beets or sugar that may be adhering to the weighing dish and glass rod; then introduce about 75 c.c. of 95 per cent alcohol into the flask, connect the apparatus in the manner shown in Fig. 52, care being taken that all the connections are tight so that alcohol will not be lost by leaking; now heat the flask in a water-bath at slow boiling and continue the extraction from one to two hours. A trace of sugar in the alcohol may be qualitatively examined by the anaphthol test which is claimed to be sensitive enough to detect 1 part of sucrose in 5000 parts of water. Remove the stopper t from the opening at the top of the syphon tube, take a few drops of the alcohol by means of a small glass tubing, transfer to a test-tube, add 2 c.c. of distilled water and five drops of a 20 per cent alcoholic a-naphthol solution, and mix the whole; now hold the test-tube in an oblique position, pour 10 c.c. of chemically pure concentrated sulphuric acid along the side of the tube so that the acid will run down to the bottom of the tube without mixing with the alcoholic solution and allow it to stand for a few minutes. In the presence of sucrose, a color, ranging from faint purple to deep violet, according to the amount of sucrose, will appear between the a-naphthol solution and the sulphuric acid. A green, yellow, or reddish color does not indicate the presence of sucrose. When the a-naphthol test indicates the absence of sucrose, disconnect the apparatus, cool the flask and contents to 17.5°C, add two or three drops of lead subacetate solution, complete the volume of the solution to 100 c.c. with water, mix thoroughly, filter, and polarize. When absolute accuracy is required, the extraction should be continued with a second flask containing 75 c.c. of 80 per cent alcohol and the polariscope

reading from the second extraction, should there be any, must be added to the first.

As was stated previously, Pellet's hot water digestion method is most preferred in the daily analysis of beets in a sugar-house laboratory, but this method should be checked from time to time with the hot alcoholic digestion or alcoholic extraction method as the composition of beets is quite variable and the hot water digestion method can not always be relied upon.

Examination of Cossette Juice for Brix Degree, Sugar, and Purity.—It is customary in the sugar-house to examine beet-juice for Brix degree, sugar, and purity, since they indicate the quality of the juice and knowledge of these matters generally gives sugar-experts important bearings in conducting the operation of the process. The sample may be obtained by pressing the remainder of the cossettes ground for direct analysis. Take the Brix degree with a Brix spindle, determine the polariscope reading by the 110 c.c. volume method, and find the sugar and purity in Table IV.

The Principle of Diffusion.—When two liquids, which are capable of dropping and which do not produce chemical change, are brought in direct contact and allowed to stand for a certain length of time, they mix uniformly without the application of mechanical impulsion or chemical force. Such action is caused by the movement of the molecules and is called Diffusion or Free Diffusion. The same action takes place even when two liquids are separated by a membrane. This is due to the porosity of the membrane, for it is simply a fine network of plant or animal tissues. Diffusion through a membrane is called Membrane Diffusion, Omose, or Dialysis. In this work, just membrane diffusion will be studied as it is the fundamental principle of the diffusion process of cossettes. Membrane diffusion may be shown by a simple experiment: fill a sac made of membrane or parchment paper (bladder answers well) with a sugar solution, surround it with water in a vessel. When allowed to stand for a certain length of time, an interchange will take place between the solution within the sac and the water surrounding it, that is,

sugar will pass out of the sac while water will penetrate into the sac through the membrane. The action will continue until the solutions within and without the sac become uniformly mix-By continuing the renewal of water indefinitely, we may be able to remove all the sugar contained in the solution within the sac. If two, three, ten, a hundred, or a thousand sacs were brought in direct contact with one another, the same action would take place through the membranes. This action will continue until the contents in all the sacs become of the same strength and an equilibrium of molecular attractions prevails in the interior of each sac, and, then, diffusion would no longer take place. When a compact group of sacs is surrounded with water, diffusion or interchange will take place, first, between the water and the outermost row of sacs, next, between the first row and the second row of sacs, then, between the second and third rows of sacs, and so on. By renewing the water from time to time, all the sugar within the sacs may be removed.

The extraction of sugar from beets is based on this membrane diffusion. As stated previously, beet consists of a great number of minute sacs which are called plant-cells. These cells are elongated, their diameter varying from 0.014 to 0.022 m.m. and their length, from 0.054 to 0.089 m.m. The cell-walls are porous and consist mainly of cellulose and pectine matter. All the cells of fresh beets contain a juice known in botany as cell-sap, which is composed of water and soluble matter. There are two kinds of soluble matter in reference to crystallizability and diffusibility through a membrane; one class is crystalloids, or substances which crystallize; the other, colloids, or substances which do not crystallize. The former pass through a membrane more readily than the latter. Sugar and salts belong to the crystalloids; albumen and amides belong to the colloids.

If we surround beet slices composed of a great number of cells, whose contents are all nearly alike, with water, an interchange of water and soluble matter will take place, first between the surrounding water and the outermost row of cells. When the composition of the contents of the first row of cells becomes

different from that of the second row, then the contents of the first row of cells behave toward those of the second row exactly the same as the water did toward the first row. Molecules of sugar and salts in the cells of the second row diffuse into those of the first row while the cells of the first row give up their sugar and saline contents to the surrounding water. In consequence of the change of composition of the contents in the cells of the second row, an interchange will take place between the second and third rows. The cells of the third row give up a part of their sugar and salt contents to those of the second, the cells of the second row, to those of the first row, and the cells of the first row, to the surrounding water, while the water travels in the opposite direction. Thus, molecules of diffusible substances will travel from one cell to another. It is evident, then, that the greater the number of cells lying upon one another and the further the cells are removed from the surrounding water, the greater would be the time required for the travel of the sugar and the salts; for example, the time required of the contents of the cells of the hundredth row to reach the surrounding water would be one hundred times that required of the contents of the cells of the first row. It is on this account that beets are sliced into thin chips so that as many cells as possible may come in direct contact with the water and diffusion be accomplished in a shorter time. There are, however, in sliced beets. about 6.4 per cent of torn and bursted cells. The contents of such cells are simply washed away.

Temperature is an important factor in the process of diffusion. Although its influence varies according to different substances, a higher temperature always causes more or less rapid diffusion. The diffusion of sugar from beet chips does not take place in cold water but begins at 50°C. The following explanation has been offered for the necessity of heating diffusion water: in the interior of cells, close to the cell-wall, there is another layer of inclosure called protoplasm. Although different in chemical composition, the structure of protoplasm is very similar to that of the cell-wall. The former, however, is much finer than the lat-

ter. A cell-wall may be compared to a coarse sieve and protoplasm, to a fine sieve. Grains that are small enough to penetrate through the sieves of the cell-wall are too large to pass through the canals in the protoplasmic sieves. The molecules of cane-sugar may be regarded as grains too large to penetrate through the canals in the protoplasmic structure. Heating kills protoplasm and changes the structure completely, that is, the protoplasmic sieves are torn up to small pieces and become so full of holes that the only obstruction the molecules of sugar have to contend with is the coarse sieve of the cell-walls. temperature of the diffusion juice should be raised to a point just enough to soften the cossettes but not to scald them; for scalding renders cossettes too soft and causes them to lie on one another, close the screens at the bottom of the diffuser, and obstruct the circulation of the juice. Another objection to too high temperature is that it causes more non-sugars to be diffused and makes the process of crystallization of sugar more difficult. suitable temperature for the diffusion process varies according to the quality of the beets; usually, it is kept within the limits between 78°C (172°F) and 85°C (185°F). When the beets are sound and the purity of the juice is high, the temperature may be kept as high as permissible. When the beets are unsound, that is, fermented, unripe, or frozen, the temperature should be kept at the lowest limit. Especially when the crystallization of sugar is slow and difficult in the vacuum-pan, the diffusion process should be carried on rapidly at the lowest temperature permissible so that, as much as possible, non-sugars may be prevented from passing into the diffusion juice.

Diffusion Apparatus.—The apparatus employed to-day for the extraction of juice from beets is called a Diffusion Battery consisting of several Diffusers. The battery commonly used consists of 10, 12, or 14 diffusers. In this article, one consisting of 12 diffusers will be briefly described, as it is the most common form. The Roman numerals I, II, III, in Fig. 53 represent diffusers. A usual form of diffuser is a cylinder ending in truncated cones provided with powerful covers. The lower cone and

cover are lined with screens so that the cossettes may not pass out of the diffuser. The lower cover is opened or closed by means of hydraulic pressure; after being closed, it is held up tight by means of a latch. Each diffuser is provided with a heating chamber called a *Calorisator*, designated in Fig. 53 by the Arabic numerals 1, 2, 3. Each diffuser is also connected with a waterpipe W and a juice-pipe J.  $a_1$ ,  $a_2$ ,  $a_3$ , represent water-valves;  $b_1$ ,  $b_2$ ,  $b_3$ , juice-valves, that is, valves used for admitting juice into the diffusers or drawing juice therefrom;  $c_1$ ,  $c_2$ ,  $c_3$ , circulation-valves, that is, valves used for cutting the diffusers from or opening them into the circulation of the juice;  $o_1$ ,  $o_2$ ,  $o_3$ , air-

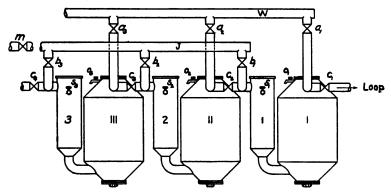


Fig. 53.—Elevation view of diffusion battery.

cocks;  $s_1$ ,  $s_2$ ,  $s_3$ , steam-valves for heating the calorisators; m represents the valve at the measuring tank. When  $c_1$ ,  $b_1$ ,  $a_2$ ,  $b_2$ ,  $a_3$ ,  $c_4$  are closed and  $a_1$ ,  $c_2$ ,  $c_3$ ,  $b_3$  are open, water will enter the diffuser I from the top, flow down to the bottom, then rise through the calorisator I; it will now enter the diffuser II from the top, rise through the calorisator 2, then flow into the diffuser III and will again rise through the calorisator 3 and then pass through  $b_3$  into the juice-pipe J. It should be noted here that, when juice or water is in circulation, all water-valves and all juice-valves are closed, excepting the water-valve through which water is admitted and the juice-valve from which the juice is being drawn. When a diffuser is filled with fresh cossettes,

it is desirable to admit the juice from the bottom of the diffuser in order to prevent the cossettes from packing and un-uniform diffusion; for, should the juice enter from the top of the diffuser, it will beat down the cossettes, cause them to pack, and the air carried with it would render the diffusion uneven. This may be accomplished, taking the diffuser I as the one to be filled with juice, by closing  $a_1$ ,  $c_1$ ,  $c_2$ , and by opening  $b_1$ , and the air-cock  $o_1$ , whereupon the juice will enter through  $b_1$  into the calorisator I from the top, flow down and enter the diffuser I from the bottom, rise through the cossettes to the top of the diffuser and flow out from the air-cock  $o_1$ , when the diffuser is full. The juice may now be drawn out from the diffuser I by opening  $c_1$  and m at the measuring tank.

Fig. 54 represents a plan-view of the whole battery consisting

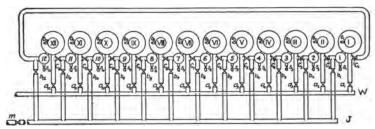


Fig. 54.—Plan view of diffusion battery.

of 12 diffusers.  $a_1-a_{12}$ ,  $b_1-b_{12}$ ,  $c_1-c_{12}$ ,  $s_1-s_{12}$ ,  $o_1-o_{12}$  represent water-valves, juice-valves, circulation-valves, steam-valves, and air-cocks, respectively; W, a water-pipe supplied with water from a tower reservoir; J, a juice-pipe connected with juice measuring tank. The valve m serves to close or open the communication between the diffusion battery and the measuring tank.

Operation of Diffusion Battery.—For starting the diffusion of cossettes, it is necessary to first prepare 3 diffusers full of hot water. After seeing that all the valves and the lower covers of all the diffusers are closed, open the water-valve  $a_9$  (Fig. 54) to admit water into the diffuser IX. When this is full, close the top cover and the air-cock  $o_9$ , open  $c_{10}$ , allowing the water to flow into X, and the steam-valve  $s_9$ , to admit steam into the steam-

chamber of the calorisator 9 so that the water passing through it may be heated. When X is full, close the cover and  $o_{10}$  and open  $c_{11}$  to admit water into XI and  $s_{10}$  to heat the calorisator 10. After the diffuser XI is filled, the diffuser XII is filled in the same manner. The flow of water and the amount of steam admitted into the calorisators should be so regulated that the temperature of the water passing out of the calorisator 12 would reach to from 87° to 90°C.

While hot water is being prepared, fill the diffusers with the cossettes, beginning with the diffuser I. When I is filled with a required amount of cossettes, close the cover, open  $b_{12}$ ,  $b_1$ , and o<sub>1</sub>, whereupon the hot water in the diffuser XII will pass through  $b_{12}$ , J,  $b_{1}$ , and flow down through the calorisator I into the bottom of the diffuser I, then rise quietly through the cossettes to the top of the diffuser. The steam for the calorisator I should be so regulated that the temperature of the water entering the diffuser I is kept between 87° and 90°C. When the diffuser I is full of water or juice, as may be indicated by the flow of the juice from the air-cock  $o_1$ , close  $o_1$  and  $o_{12}$  and open  $o_1$ , whereupon the flow of the juice in I is reversed, that is, the juice flows down through the diffuser I and rises through the calorisator I. When the diffuser II is filled with cossettes, close the cover, open  $b_2$  and  $o_2$ , whereupon the juice in diffuser I will pass through  $b_1$ , J,  $b_2$ , flow down through the calorisator 2, then rise through the cossettes in the diffuser II. The valve s2 should also be opened as soon as  $b_2$  is opened so that the juice passing through the calorisator 2 may be heated. When II is full of juice, close  $b_1$  and  $o_2$  and open  $c_2$ ; if the diffuser III is already filled with cossettes, close the cover and open  $b_3$ ,  $s_3$ , and  $o_3$ , whereupon the juice heated through the calorisator 3 will flow into the diffuser III in the same manner as it did into the diffuser II. The remainder of the diffusers may be filled with cossettes and juice in the same manner.

As may be judged from the manner the juice circulates, the density of the juice becomes higher and higher as it flows from one diffuser to another. When it passes through a certain num-

ber of diffusers, its density becomes the same as that of the normal juice, that is, the juice expressed from the beets. tomary to draw the juice from the diffusers as soon as the density becomes 0.8 that of the normal juice; for instance, when the density of beet-juice averages 16.0° Brix, the density of the juice to be drawn from a diffuser should be about 16.0° × 0.8=12.8° Brix. This density, however, may not necessarily be attained in starting but as soon as the battery is running regularly, it should be so regulated as to attain such density as specified. juice may be drawn from a diffuser in the following manner: assuming that the diffuser VII has just been filled with cossettes and juice and the juice is to be drawn therefrom, close  $b_a$  and  $o_7$  and open  $c_7$  and the valve m, whereupon the juice will be forced into the measuring tank by the pressure of the water supplied from the tower reservoir. The measuring tank is located between the diffusion battery and the saturation-tank. It is an ordinary tank graduated in hectoliters by means of a floating gauge. The volume of the juice to be drawn is variable according to the circumstances attending the operation. It is, however, customary to draw about 10 hectoliters from each ton of beets freshly added; for instance, should a diffuser be filled with 3 tons of fresh cossettes, about 30 hectoliters of juice should be In case the exhaustion of cossettes is insufficient, more juice should be drawn; should the exhaustion be carried too far, it is necessary to reduce the amount of the juice to be drawn. When a desired volume of the juice is drawn from the diffuser VII, close the valve m. If the diffuser VIII is already filled with cossettes, open  $b_8$ ,  $s_8$ , and  $o_8$  so that the hot juice may flow therein; when it is full, close  $o_8$  and  $b_7$  and open  $c_8$  and  $m_8$ whereupon the juice will be forced into the measuring tank again. The juice may be drawn from any diffuser in the same manner.

Diffusers IX, X, XI, and XII which are full of hot water must be emptied so that they may be filled with cossettes and juice. In emptying the diffuser IX, first open  $a_{10}$  so that the water pressure in the battery may be uninterrupted, then cut out the dif-

fuser IX from circulation by closing  $a_0$  and  $c_{10}$ ; now open  $o_0$ , remove the top cover and the latch from the bottom cover, close the hydraulic pressure valve and open the hydraulic pressure release valve, whereupon the hydraulic pressure will be removed, the bottom cover be lowered, and the contents of the diffuser be emptied. When the diffuser is empty, close the bottom cover by closing the hydraulic pressure release valve and by opening the hydraulic pressure valve, whereupon the cover will be raised, and then lock it with the latch. The diffuser is now ready to be filled with cossettes and juice. The diffusers X, XI, and XII may be emptied in a similar manner.

As stated previously, the density of the juice becomes higher and higher as it flows through more and more diffusers. other hand, it becomes lower and lower in density, following the diffusers in the direction opposite to the flow of the juice, and finally it becomes nearly the same as water in the diffuser into which water is admitted. By the time the juice is being drawn from the diffuser XI and the diffuser XII is being filled with fresh cossettes, the sugar content of the cossettes in the diffuser I becomes so sufficiently low that the diffuser may be emptied. In emptying the diffuser I, open a<sub>2</sub> in order to continue the water pressure in the battery, cut out the diffuser I from the juice circulation by closing  $c_2$  and  $a_1$ , then open  $a_1$ , remove the top cover and the latch from the bottom cover, close the hydraulic pressure valve and open the hydraulic pressure release valve, whereupon the bottom cover will be lowered and the contents of the diffuser be emptied. Some of the exhausted cossettes adhering to the sides and the bottom of the diffuser may be washed away by opening  $a_1$  for a few seconds. When the diffuser is clean, close the bottom cover by closing the hydraulic pressure release valve, opening the hydraulic pressure valve, and replacing the latch. It is now ready to be refilled with cossettes and juice. While the juice is being drawn from the diffuser XII, the diffuser II may be emptied in a similar manner. These operations of filling the diffusers with cossettes and juice, drawing the juice and emptying the diffusers may be continued indefinitely.

Temperature.—In order to carry out the diffusion process successfully, the temperature in each diffuser should be carefully regulated by means of a steam valve belonging to the calorisator, according to regulations established by long experience. The quality of beets requires a variation of temperature and therefore it is necessary to find a suitable temperature for each case. Dupont recommends the following temperature in a battery of 12 diffusers, 10 of which are in circulation:

Diffusers 1, 2, 3, 4, 5, 6, 7, 8, 9, 10.
Temperature C 40 60 76-85 65-70 40-50
Temperature F 104 140 169-185 149-158 104-122.

No. I represents any diffuser into which water is admitted; No. 10, the diffuser from which the juice is being drawn.

Schultz recommends the following regulation:

Diffusers 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, Temperature C 10 20 30 40 55 70 75 50 40 20 filling emptying. Temperature F 50 68 86 104 131 158 167 122 104 68

When the operation is to be slowed down, the temperature should be lowered suitable to the speed, as a high temperature in slow operation is liable to diffuse an excess of non-sugars. In case the operation is to be interrupted for a short time, all the steam valves should be closed.

Emptying the Entire Battery.—After an operation for a certain number of days, most of the machinery needs cleaning and some repairing and it is necessary to empty the entire battery and stop the operation until the cleaning and necessary repairings are done, which usually occupy about 12 hours. Assume that 10 diffusers are in juice circulation at the time emptying is to be begun, water being admitted into the diffuser III and the diffuser XII being just filled with cossettes and juice. Draw the usual volume of the juice from the diffuser XII and empty the diffuser III in the usual manner and wait for about the interval of time required for filling a diffuser with cossettes and juice; then draw the usual volume of the juice from the diffuser XII again and empty the diffuser IV. Continue this until the juice drawn does not contain more than 0.5 per cent of sugar. may usually be attained when the diffuser VIII is emptied. The remainder of the diffusers may be emptied without drawing the juice, since it is so dilute that it could not be profitably worked.

Chemical Analysis of Diffusion Juice, Exhausted Cossettes (Pulp), and Waste Water.—The volume of the juice to be drawn and the temperature in the diffusers should be controlled largely by chemical analysis; for when the density of the juice and the sugar content in exhausted cossettes are higher or lower than the regulation requires, as shown by chemical examination, it is possible to correct them by regulating the temperature and varying the volume of the juice to be drawn.

A small portion of the diffusion juice should be saved in a sample pail every time the juice has been drawn from a diffuser, in order to obtain an average sample during a specified interval of time, which is usually about two hours. The Brix, sugar, and purity should be determined every two hours. Mix the sample thoroughly, transfer to a glass cylinder, cool down to approximately 17.5°C in cold water, mix again, allow it to stand until the air-bubbles are expelled from the juice; then take the Brix degree, correct it for temperature if necessary, determine the polariscope reading by the 110 c.c. volume method and find the sugar and the purity in Table IV.

The sugar content in exhausted cossettes may be best determined by the hot water digestion method; but for daily control work, indirect analysis by the 110 c.c. volume method is employed, as it is the quickest and fairly reliable. Small portions of pulp and waste water should be saved from each diffuser emptied and be analyzed every hour. Grind the sample of pulp through a sausage-grinder, express the juice, determine the polariscope reading by the 110 c.c. volume method, omitting the Brix degree as it is usually low enough to be disregarded. The polariscope reading multiplied by 0.28 will give approximately the per cent of sugar in the pulp. The sugar content in the waste water may also be determined by the 110 c.c. volume method, omitting the Brix degree, and the polariscope reading multiplied by 0.28 will give the per cent of sugar in the waste water.

The sugar content in the pulp and waste water may be conveniently found in the following table:

	.0	.ı	.2	•3	-4	-5	.6	-7	.8	.9
0	.00			.09						.25
I	.29	.32	.35	.38	.40	.43	.46	.48	.51	∙54
2	∙57	.60	.63	.66	. <b>6</b> 8	.71	• •74	.76	.79	.82
3		.88	.91	-94	.96	.99	1.02	1.04	1.07	1.10
4	1.14	1.17	1.20	. 1 <b>.23</b>	1.25	1.28	1.31	1.33	1.36	1.39
5	1.42	1.45	1.48	1.51	1.53	1.56	1.59	1.61	1.64	τ.67

The numbers in the first column on the left are integral numbers for the polariscope reading; those in the first horizontal row on the top are decimal numbers for the polariscope reading. The use of the table may be shown by an example: assuming the polariscope reading to be 2.4, the per cent of sugar in the pulp or waste water will be found in the column under .4 and opposite 2. The number sought is .68.

The operation of the diffusion battery should be so regulated that the sugar content in the pulp does not exceed 0.50 per cent and that in the waste water, 0.20 per cent.

Calculation of Loss of Sugar in Pulp and Waste Water In Per Cent to the Weight of Beets .- According to Stammer, the pulp as it goes out of the diffusers amounts to about 80 per cent and the waste water, to 150 per cent of the weight of beets. It is evident, then, that the per cent of sugar in the beets lost through pulp would be the per cent of sugar found in the pulp multiplied by 0.80 and that the per cent of sugar in the beets lost through waste water would be the per cent of sugar in the waste water multiplied by 1.50. For example, when 0.45 per cent of sugar is found in the pulp, the per cent of sugar in the beets lost through pulp would be 0.45 × 0.80 = 0.36; when 0.17 per cent of sugar is found in the waste water, the per cent of sugar in the beets lost through waste water would be 0.17×1.50=0.255. The total loss of sugar in the beets through pulp and waste water would, then, be 0.36+0.255=0.615. This is practically the same as the sum of the per cents of sugar found in the pulp and waste water, that is, 0.45+0.17=0.62; hence, in common practice. the sum of the per cents of sugar found in the pulp and waste water is taken for the per cent of sugar in the beets lost through pulp and waste water.

Disposition of Exhausted Cossettes.—The exhausted cossettes emptied from the diffusers are carried away by means of a chain conveyor, passed through powerful presses in order to remove the excesses of water. Unpressed pulp contains about 5.5 per cent of dry substance; pressed pulp contains about 8.5 per cent of dry substance. Pressed pulp is usually siloed and used for feeding stock. In some factories, the pulp is dried in a drying machine and sent to market as dry pulp fodder.

Composition of Pulp.—The composition of the pulp is very variable. It contains, some years, more fibers and nitrogen compounds, some years, less. The variation of the composition of the pulp also depends upon the manner of treatment of the beets in the factory, that is, the composition of the pulp varies according to the degree of fineness, temperature, and the manner of pressing. Maecher gives the following average analytical results for fresh, fermented, and dry exhausted cossettes:

	Fresh per cent.	Fermented per cent.	Dry per cent.
Albumen	.89	1.07	6.54
Crude fibre	2.39	2.80	18.57
Fat	.05	.11	
Nitrogen free substance	6.32	6.41	56.29
Ash	.58	1.09	6.02
Water	89.77	88.52	12.58

Principle of Defecation of the Diffusion Juice with Lime.—Several reagents have been proposed for the defecation of beet-juice, but lime is most commonly adopted owing to its inexpensiveness and non-poisonous character. The action of lime on the juice is three-fold: I, Sterilization; 2, precipitation; 3, decomposition.

In the beet-juice, there are fungus germs of every kind, which were introduced from the soil, water, air, and, sometimes, diseased or decomposed beets. These fungus germs cause fermentation and putrefaction of the juice. When the juice is treated with lime under high temperature, however, all these fungus germs are killed, hence, the process may be called the sterilization of the juice.

Beet-juice contains impurities which could be removed or be

made less objectionable to the crystallization of sugar by treating with lime. There are, in beet-juice, many organic acids whose calcium salts are insoluble or difficultly soluble in water, such as oxalic acid, tartaric acid as discussed under Constituents of Non-sugars in Beet-Juice. These compounds may be removed wholly or partially by treating with lime. The inorganic compounds that may be completely removed from the beet-juice by the action of lime are phosphoric acid, magnesia, and silicic acid.

The organic compounds, constituting beet-juice, which could not be precipitated but may be decomposed by the action of lime are albumen, invert sugar, and amides. Albumen is partly precipitated by heating the diffusion juice, but the greater part thereof remains dissolved in the juice. When the juice is treated with time, the soluble albumen is decomposed first into propeptone and peptone and later into amido-acids, such as leucine, tyrosine, aspartic acid, and so forth. Propeptone and peptone are uncrystallizable but amido-acids and their salts are crystallizable. Uncrystallizable compounds render the crystallization of sugar difficult and are called commonly melasgenic.

When treated with lime, invert sugar, like albumen, is readily converted into crystallizable compounds such as lactic acid and saccharic acid. When treated with lime, amides such as asparagine and glutamine dissociate into amido-acids and ammonia. The principal source of the development of ammonia from beet-juice is the decomposition of amides. According to Reiset, one liter of beet-juice liberates an average of 0.634 grams of ammonia.

The coloring matter in beet-juice may be removed partially by treating with lime. Coloring matter in beet-juice is known as *Chromogen*. It is, originally, or in the cells of beets, colorless and is called, by Reinke, *Rhodogen*. When exposed to air, chromogen becomes oxidized, acquires quite a dark color and is called, by Reinke, *Betarot*. Neither lime nor boneblack has the power to remove chromogen. Betarot, however, is separated from the juice by treating with lime. The chromogen which remains in the juice, through oxidation or decomposition during the latter part of the process, acquires a dark color. The dark

color of thick juice, fillmass, and syrups is said to be due to the presence of this product.

Another important action of lime on beet-juice is a mechanical one. Particles and filaments of beets and substances precipitated by heating are all suspended in the diffusion juice. All these matters are carried down with the precipitate of lime.

Apparatus for Liming Raw Juice.—The liming of a raw diffusion juice is usually done in an ordinary tank provided with an agitator for mixing the juice and lime. It is also provided with a steam coil and a thermometer for controlling the temperature of the juice. This apparatus is called a Raw Juice Mixer or a Defecator. In a factory where milk of saccharate or milk of lime is used for the defecation of juice, a small funneltank for measuring the milk of lime is located over the defecator. In some factories, liming is carried out in a saturation-tank immediately before saturation.

Forms of Lime Applied in Defecation.—Lime is commonly applied in the defecation of raw juice in one of the three fol-1, slaked lime; 2, dry powder; 3, saccharate. lowing forms: The application of lime in the form of slaked lime is the simplest. In slaking lime, a desired quantity of lime in lumps is placed in a tank, covered with water and stirred continually by means of an agitator until most of the lime is slaked, forming a milky liquid, which is commonly called Milk of Lime. Water is added to the liquid until a Baumé spindle indicates about 20° at which the liquid contains about 17.72 per cent of lime (CaO). When ready, it is pumped up to the raw juice mixer. Lime powder is made by crushing burnt limestone in a powerful crusher, grinding it fine in stone mills and sifting it in bolters. Powdered lime is conveyed to the defecation-tank by means of a scroll. The preparation of saccharate of lime will be described under The saccharate of lime obtained in STEFFEN'S PROCESS. Steffen's process is diluted with sweet water (the wash water from the first and second saturation juice filter presses) until a Baumé spindle indicates 16° or 17° and applied for the defecation of raw juice.

Quantity of Lime to be Applied.—The quantity of lime to be

used for the defecation of raw juice with satisfactory results ranges between wide limits. From 0.15 to 0.20 per cent of lime to the weight of the juice is sufficient to neutralize acids and to precipitate all the precipitable substances present in the beetjuice. From 0.50 to 0.75 per cent of lime to the weight of the juice is sufficient to produce a clear juice. With such quantities as cited above, however, filtration of the saturated juice is very slow and the operation on a large scale could not be carried out. In common practice, from 2.5 to 3.0 per cent of lime to the weight of beets is applied. When the purity of the juice is very low, still a larger quantity of lime may be applied, as high as 5 per cent of lime to the weight of beets. One hectoliter of milk of lime at Baumé 20° contains 20.6 kilograms of lime, hence an application of I hectoliter of milk of lime at Baumé 20° to every 10 hectoliters of the juice will amount to about 2.5 per cent of lime to the weight of beets. In a factory where Steffen's process of extracting sugar from molasses is in operation, about 5 per cent of lime to the weight of beets may be used, as a large amount of lime is usually available in a form of saccharate. An application of I hectoliter of milk of saccharate at Baumé 16° to every 3 hectoliters of raw juice usually brings about this proportion. .

Temperature for Defecation.—Another important factor in the defecation of the juice with lime is heating during defecation. The most favorable temperature for the defecation of the juice of normal beets is from 70° to 85°C (158° to 185°F). In case the purity of the beet-juice is low, the temperature may be raised to as high as 90°C (194°F). A temperature above this point, however, is not advisable, for a considerable quantity of the tri-calcium saccharate is liable to be formed at such a high temperature. At any rate, the temperature of the juice should be raised sufficiently high to decompose a greater part of the nitrogenous compounds and the invert sugar. Cold defecation is advocated by some sugar-experts; but it is not practical as defecation and filtration are very slow at low temperature.

The duration of defecation varies according to the temperature adopted. At a temperature between 70° and 80°C (158°-178°F), defecation should last about 15 minutes; at a higher temperature, from 5 to 10 minutes.

Operation of Defecator.—Draw about 90 hectoliters of raw juice from the measuring tank into the defecator, heat up to about 80°C (178°F), introduce about 9 hectoliters of milk of lime at Baumé 20°, or 30 hectoliters of milk of saccharate at Baumé 16° from the funnel-tank and mix continually, keeping the temperature at about 85°C (185°F). At the end of about 10 minutes, empty the mixture into the carbonatation or saturation tank.

Principle of Saturation or Carbonatation.—As stated previously, the raw juice is treated with a large excess of lime. It is a common practice to remove this excess of lime by precipitating with carbon dioxide gas, as an excess of lime keeps sugar in the form of insoluble saccharates. It is also claimed that carbon dioxide gas improves the quality of the juice otherwise than the precipitation of lime, since it precipitates the mineral and organic salts of lime remaining in the juice. Another very beneficial action of carbon dioxide gas is that it renders the precipitates of lime compounds granular and causes the juice to filter readily. During saturation, many changes take place. The first product is a complex combination of calcium carbonate, calcium saccharate, and calcium oxide which is called sucrocarbonate of lime. Sucrocarbonate is gelatinous and insoluble in water. On further heating and saturation, this compound is decomposed, lime being separated in the form of calcium carbonate and sugar being set The temperature should be raised to from 88° to 90°C (190° to 194°F) as the juice improves best at a higher temperature. Stohmann encourages boiling the juice at the end of satura-Classen recommends passing the saturated juice through a reheater heated up to 100°C (212°F).

The lime content in the juice after a saturation is called Alka-linity. The degree of alkalinity is a very important factor in the treatment of the juice. An over-saturation dissolves, to some extent, the coloring matter and other impurities, rendering the

juice dark-colored and also otherwise undesirable. Too high alkalinity causes sugar to remain in the form of insoluble saccharates and renders the loss of sugar through lime cakes too high. Too high alkalinity also makes filtration of the juice difficult. The most suitable alkalinity at the end of the first saturation is 0.100 per cent, at the end of the second saturation 0.03 per cent, using phenolphthalein as the indicator.

Saturation Gas.—Saturation gas is drawn from the top of the lime-kiln through a gas-washer and forced into the saturationtank by means of a pump. Lime-kiln gas usually contains from 25 to 35 per cent of carbon dioxide. In order to obtain the best regulation, the saturation gas should be tested every hour. The simplest method for testing saturation gas is Stammer's. special pipette of 100 c.c. capacity has been prepared for this test. It is simply a burette graduated in tenths with a glass cock at one end and a zero mark at the open end. Testing may be carried out as follows: connect the glass cock end of a 100 c.c. pipette with a pet cock attached to a gas-pipe by means of rubber tubing, pass the gas into the pipette for one minute, keeping the open end of the pipette under the surface of water in a pail, and close the cock; then hold the pipette vertically at such a height that the zero mark comes even with the surface of the water, open the cock so that the water within the pipette may rise and close the cock as soon as the water within the pipette Now introduce a small piece of causreaches the zero mark. tic soda into the pipette through the open end while under water, close the open end with a finger, remove the pipette from the water and shake vigorously, place the open end of the pipette under water again and loosen the finger so that water may be admitted into the pipette to fill a space caused by the absorption of carbon dioxide by caustic soda. Repeat shaking the pipette and admitting water therein several times. When no more water can be admitted, remove the finger under water, hold the pipette at such a height that the level of the water is the same within and without the pipette and take a reading which will give the per cent of carbon dioxide directly. In case a 50 c.c. pipette is used, the reading must be multiplied by 2.

Saturation or Carbonatation Tank.—Fig. 55 shows the construction of a saturation tank. It is a rectangular tank provided

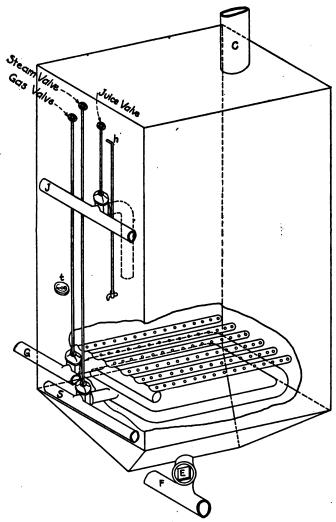


Fig. 55.—Saturation tank.

with steam-coils and gas-tubes. G represents the gas-pipe; J, the juice-pipe; S, the steam-pipe; t, the thermometer; h, the

sample-cock; C, the chimney. The gas-valve admits carbon dioxide gas into the perforated gas-tubes which discharge the gas into the juice. The juice-valve admits the limed juice from the defecator into the saturation tank. The steam-valve admits steam into the steam-coil which is often perforated. E represents the outlet cock which discharges saturated juice into the pipe F, which in turn carries the juice to a saturation juice storage tank.

Operation of First Saturation Tank.—As soon as the tank is filled with about 90 hectoliters of the juice and 30 hectoliters of milk of saccharate or 9 hectoliters of milk of lime, which were already mixed in a defecator, close the juice-valve, open the gasvalve, and continue saturating until the alkalinity of the juice A point at which the saturation is reduced to 0.100 per cent. is to be terminated should be watched very carefully as oversaturation injures the quality of the juice. After a little practice, a carbonator may be able to judge when the gas is to be cut off from the appearance of the saturated juice. Draw a sample of the saturated juice, from the sample-cock, in a sample cup, transfer to a glass beaker, and watch it in a strong light. Sampling should be done frequently in order to avoid oversaturation. When the saturation is carried to a proper point, the precipitate of lime will appear granular and separates from the juice quickly. Then close the gas-valve and test the alkalinity with a standard acid solution in order to be sure that a desired point in the alkalinity is nearly reached. Should it be higher than 0.100 per cent, open the gas-valve for a few seconds and test the alkalinity again. Continue this process until the alkalinity reaches to about 0.100 per cent. It is sometimes desirable to vary the alkalinity, for instance, when the saturated juice filters very slowly or combined sugar in lime cakes is unusually high, the alkalinity of the juice should be as low as it could be made without injuring the juice. When the juice is over-saturated, some more milk of lime or saccharate should be added and saturated to a proper alkalinity. When the alkalinity of the juice is brought down to a desired point, heat it up to about

88°C (190°F) and open the cock E in order to let the juice flow into the saturation juice storage tank.

Testing Alkalinity of the Juice.—For testing the alkalinity of the juice, a standard sulphuric acid solution of such strength that

1 c.c. would neutralize 0.001 gram of calcium oxide 
$$\left(\frac{N}{28}$$
 solution  $\right)$ .

Testing may be conducted as follows: filter a sample of the saturated juice through a filter-paper, collect the filtrate in a beaker, measure exactly 10 c.c. thereof by means of a 10 c.c. capacity tin cup, transfer to a flat bottomed white porcelain dish, add a few drops of phenolphthalein solution whereupon the juice will be colored pink. Now fill a 25 c.c. automatic filling burette

(Fig. 43) to exactly the zero mark with the  $\frac{N}{28}$  sulphuric acid

solution just cited, run the acid into the juice a little at a time stirring continually until the pink color just disappears and read the burette. When the burette reads 10 c.c., it indicates that 10 c.c. of the juice contains 10×0.001=0.01 gram of calcium oxide. When expressed in per cent, the lime content would be

 $\frac{0.01 \times 100}{10}$  = 0.100 per cent. Should 10 c.c of the juice require

9 c.c. of the acid, the alkalinity of the juice would be  $\frac{0.009 \times 100}{10}$ 

=0.090 per cent. It is evident from this that when 10 c.c. of the juice is titrated with the acid solution, 1 c.c. of which neutralizes 0.001 gram of lime, the percentage of alkalinity of the juice may be obtained by dividing the burette reading by 100. In this test, the per cent of alkalinity has been determined in a certain volume but not in a weight of juice, hence, it may be called volume per cent. When it is desirable to obtain a true per cent, the volume per cent should be divided by the specific gravity of the juice. A portion of the juice filtered for the final test of the alkalinity should be saved from every tank saturated in order to obtain an average sample. The average sample should be taken to the chemical laboratory once every hour and be accurately tested by a chemist and recorded in the laboratory re-

port book. Alkalinity is variable according to the indicators employed. For example, alkalinity is higher when phenolphthalein is used than when methyl orange is used. In this work, phenolphthalein alkalinity is used throughout.

Filtration of First Saturation Juice.—The first saturation juice is mixed with a large bulk of precipitate of calcium carbonate which must be rid of before it can be subjected to the second saturation, for the alkalinity of the juice could not be reduced much below 0.100 per cent in the presence of the precipitate from the first saturation without redissolving the coloring matter and some of the other impurities.

Filter Press.—A filter press is a collection of cast-iron plates and frames. In Fig. 56, (a) represents a plate; (b), a frame.

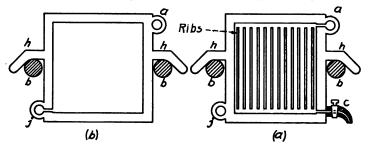


Fig. 56.—Filter press frame and plate.

The plates are provided with ribs on both sides excepting the two end plates which are ribbed just on one side. a is a passage for water; j, that for juice; h, a handle by which the plates are resting on a horizontal bar b. a, j, h, and b in the frame correspond to those of the plates. When in use, the plates are covered with filter-cloth and the frames are pressed tightly against them. One press consists of 64 frames and 65 plates including the two heavy end plates. They are pressed tight from one end of the press by means of a screw. When they are pressed together, the a's form a passage for water; the j's, a passage for juice. The j's in the frames have openings toward the interior for admitting juice into the frame. The a's in the frames and the j's in the plates have no openings toward the interior. The plates are of two kinds; one is called water-plates, the other, juice-

plates. The a's in the water-plates have openings toward the interior constructed in such a manner as to lead the water into the channels between the ribs on both sides of the plates as shown by (a) in Fig. 56, while the a's in the juice-plates have no openings toward the interior. The plates are provided with cocks as indicated by c in (a), Fig. 56. The cocks for the waterplates are long-stemmed while those for the juice-plates are short-stemmed. The plates and frames are arranged in the following order where J, F, and W represent juice-plate, frame, and water-plate respectively: J-F-W-F-J-F-W-F-J and so on. When the water-valve is closed and the juice-valve and all cocks are open, the juice will fill the frames, filter through the cloth, flow down along the channels on the water- and juice-plates and then pass out through the cocks. When the frames are filled with the deposits of lime precipitates, the juice-valve and all water-plate cocks are closed and the water-valve is opened; the water will then spread on both sides of the water-plates along the channels thereon, penetrate through the filter-cloth and lime deposits in the frames, flow down along the channels on the juice-plates and pass out through the juice-plate cocks.

Operation of Filter Presses.—Cover all the plates properly first with jute and then with duck and close the press by pressing the frames and plates from one end of the press by means of a screw tightener. Then close the water-valve, open all the cocks and juice-valve and pump the saturation juice from the storage tank to the press, whereupon the juice will fill the press. and the clear juice will flow out through the cocks into a trough. leaving lime precipitates within the frame. The filtered juice is clear and slightly yellow. The alkalinity of this juice should be tested at least once every hour. Filling should be continued until a pressure gauge inserted in the juice-pipe indicates about 3 atmospheric pressure or 45 pounds. When the press is filled to a desired pressure, close the juice-valve and all the waterplate cocks and open the water-valve, whereupon the water will pass through the lime deposits within the frames carrying away the sugar which is adhering to the deposits. It is best to use, for washing, hot water having a temperature, if possible, corresponding to that of the juice, which is about 71°C (160°F). It is said that, when the lime deposits are washed with cold water, there will be formed sucrocarbonates of lime, which are insoluble in water, remain with the lime deposits, and help to increase the loss of sugar. Too hot water is also objectionable for washing, as it shortens the life of filter-cloth and also tends to form insoluble calcium saccharates.

The filtered juice is run directly into the second saturation tank. The first part of the wash water is also run into the second saturation tank. The latter part of the wash water is stored in a separate tank to be used for preparing milk of lime or milk of saccharate. The duration of washing varies depending upon the quality of the lime precipitate. Usually it takes from 20 to 30 minutes to wash one press. A Brix spindle graduated at 160°F may conveniently be used for regulating the washing. regulation, the wash water is usually run into the juice until the spindle indicates 5 per cent, then it is run into the wash water storage tank until the spindle indicates 2 per cent, at which point washing is terminated by closing the hot water valve. When the washing is completed, unscrew the screw tightener, pull the plates and frames apart and dump the lime deposits into the hopper which directs them into a trough below. These lime deposits are called "Lime Cakes." The lime cakes received in the trough are forwarded by means of a scroll into a mixer where they are mixed with water and converted into a cream, and then pumped into a pipe which conducts the cream into the "lime pond."

Difficulties in Filtration.—Lime cakes of normal quality appear yellowish gray and are brittle. Juices which yield such cakes are easily filtered. Sometimes, lime cakes appear greenish yellow and are slimy. The juice leaving such cakes is very difficult to filter and sugar adhering to the cakes can not be readily washed out. Difficult filtration and washing are usually caused by insufficient lime or defective saturation or heating. A poor quality of beets and the presence of an abnormal quantity of silica or iron oxide in lime are also frequently causes of bad

filtration and washing. It is said that, in the presence of a large quantity of silica, a lime cake soon becomes hard, forming an impervious slab of artificial stone. It is also said that, when iron oxide and pectine matter are present in large quantities, a flocculent and spongy mass of ferropectine is formed and obstructs the passage of juice and water through the filter-cloth. The addition of a proper quantity of lime, proper saturation and heating, careful control of the diffusion process and the proper selection of limestone generally prevent difficulties in filtration and washing.

Sugar in Lime Cakes.—It is very important to keep the sugar-house management informed of the sugar content in the lime cakes in order to compute the loss of sugar through the lime cakes. It is a practice in all sugar-houses to compute the average loss of sugar through the lime cakes in per cent to the weight of beets at the end of each period or campaign. It is also important to know the sugar content in the lime cakes as the operations in diffusion, saturation, and filtration may be guided thereby to a large extent.

Sugar may be found in lime cakes in a free state and in combination with lime. A presence of a large quantity of free sugar may be due to inattention on the part of the filter press man or to difficulties in washing the lime deposits caused by defective defecation, saturation, or heating, or by the presence of objectionable substances, such as silica, iron oxide, or pectine matter. Sugar found in combination with lime is in the form of tri-calcium saccharate or sucrocarbonate of lime. The presence of a large quantity of these combinations is produced usually by improper saturation and heating. Tri-calcium saccharate is usually the product of excessive heating while sucrocarbonate of lime is formed by insufficient saturation and heating, or by washing the deposits with cold water. It is evident from these statements that the estimation of free and combined sugar gives some bearings in regulating the operations in diffusion, defecation, saturation, heating, filtration, and washing.

Sampling Lime Cakes.—The sample of lime deposits should

be so taken that the result of the analysis thereof would nearly represent the average sugar content in the lime deposits of a whole press. The sugar content in lime cakes varies in different frames owing to the difference in the permeability of filter-cloths. It is a practice to take a sample from several frames at different places covering a whole press in order to obtain a representative sample of that press. The sugar content also varies even in the same frame, being highest near the juice-line and lowest near the water-line. It is, therefore, advisable to take a small portion of lime cake from the center of frame which usually gives approximately an average sugar content for a whole frame.

Preparation of Normal Solution from Lime Cakes.—In estimating sugar in lime cakes, the polariscope reading is made with normal solution in a 200 m.m. observation tube. A normal solution of lime cake, however, could not be prepared by making up one normal weight thereof to 100 c.c. in a 100 c.c. flask with water since the precipitate of calcium carbonate, which is the chief constituent of lime cake, occupies some space within the flask employed. Lime cake contains about 50 per cent of calcium carbonate, which has a specific gravity 2.9; hence, 50 grams of lime cake contains 25 grams of calcium carbonate

and the volume occupied thereby would be  $\frac{25}{2.9} = 8.62$  c.c.

When 50 grams of lime cake are made up to 200 c.c. with water, the volume occupied by the solution would be 200-8.62=191.38 c.c. As stated previously, a solution of normal strength is prepared by making up 26.048 grams to 100 c.c., or 52.096 grams to 200 c.c. of solution. It is evident, then, that the weight of lime cake to be made to a solution of normal strength in a 200 c.c. flask, that is, to be made up to 191.38 c.c. would be  $52.096 \times$ 

 $\frac{191.38}{200}$  = 49.85 grams or approximately 50 grams. Hence in

preparing normal solution from lime cakes, 50 grams of the sample are made up to the graduation mark in a 200 c.c. flask.

Estimation of Free Sugar in Lime Cakes.—Break off a small portion from each lump of lime cakes composing the sample,

place them all together in a mortar, mix, and beat them until the whole is uniformly mixed and becomes very soft or pasty. Weigh out 50 grams in a sugar-dish, add a little water, mix with a glass rod until the sample becomes a perfect cream, transfer it to a 200 c.c. flask completely by washing the sugar-dish with water, add 10 c.c. of dilute lead solution, complete the volume to the 200 c.c. mark with water, mix thoroughly by shaking, filter, and polarize in a 200 m.m. tube. The polariscope reading gives the per cent of sugar directly.

Estimation of Combined Sugar in Lime Cakes.—In estimating combined sugar, it is necessary to add some reagent to liberate the sugar from saccharate of lime so that it may be in solution. The reagent commonly employed is acetic acid or animonium nitrate. By treating insoluble saccharate of lime with these reagents, the following reactions take place:

Tri-calcium Acetic Calcium Sugar Water saccharate acetate  $C_{12}H_{22}O_{11}3CaO + 6CH_{5}COOH = C_{12}H_{22}O_{11} + 3(CH_{5}COO)_{2}Ca + 3H_{2}O$ Tri-calcium Ammonium Calcium Ammonia Sugar accharate  $C_{12}H_{22}O_{11}$  3 CaO + 6  $H_4NNO_3 = C_{12}H_{22}O_{11} + 3 Ca(NO_3)_2 + 6 H_3N + 3 H_2O$ . It is a common practice to determine the total sugar and take the difference between the free sugar and the total sugar for a combined sugar. In case acetic acid is to be employed for the decomposition of saccharate, after the sample is prepared, weighed, and transferred to a 200 c.c. flask, add acetic acid a little at a time, shaking continually until the color of the mixture turns distinctly darker. Some chemists use a few drops of phenolphthalein solution as an indicator, adding acetic acid until the pink color just disappears. Having thus neutralized caustic lime, add about 10 c.c. of dilute lead solution, make up to the 200 c.c. mark with water, mix, filter, and polarize in a 200 m.m. tube. When ammonium nitrate is to be used for the decomposition of saccharate, add about 5 grams of the solid substance to a weighed sample of lime cakes in a sugar-dish, mix to a perfect cream, transfer to a 200 c.c. flask, add 10 c.c. of dilute lead solution, make up to the 200 c.c. mark with water, mix, filter, and polarize in a 200 m.m. glass observation-tube, as ammonia

dissolves copper from the brass tube and gives to the solution a blue color which makes the observation difficult. The polariscope reading gives the per cent of sugar in lime cakes directly.

Second Saturation.—As stated previously, the reduction of the alkalinity of the juice in the first saturation has been limited to about 0.100 per cent as further reduction tends to dissolve coloring matter and some of the other impurities. This degree of alkalinity, however, is too high for concentration of the juice, since lime deposits on the concentration apparatus and renders them ineffective in a short time. It is a common practice to subject the juice to a second saturation, using carbon dioxide gas. process is called the Second Saturation or Carbonatation. The second saturation may be carried out with or without an addition of lime. When the purity of the juice is low, it is advisable to use some lime. From 3 to 5 hectoliters of milk of lime or milk of saccharate would be sufficient for 90 hectoliters of the juice. The second saturation not only reduces the alkalinity but improves the juice, the same as the first saturation, though in a smaller degree. The tanks used for the second saturation are constructed exactly the same as those for the first saturation. The saturation may be conducted as follows: allow the juice from the first saturation juice filter presses to flow by gravitation into a second saturation tank until the juice will run out of the sample-cock, close the juice-valve, introduce 3 or 4 hectoliters of milk of lime or milk of saccharate, heat up the juice to about 80°C (176°F), open the gas-valve for about 3 minutes, take a sample, filter, and test it in the same manner as in the first saturation. The gas-valve should be kept closed while the sample is being The alkalinity of the second saturation juice should be about 0.03 per cent. Should it be still above 0.03 per cent, open the gas-valve for a few seconds and test the juice for alkalinity again. Continue the operation until approximately 0.03 per cent of alkalinity of the juice is attained, then heat up the juice to boiling in order to break up acid calcium carbonate formed during saturation and to keep the influence of the lime constantly active. Great care must be taken not to under- or over-saturate

the juice. When the saturation and heating are completed, the juice is pumped directly from the saturation tank to the second saturation juice filter presses. The filtration of the second saturation juice is conducted in the same manner as of the first saturation juice. The juice from the second saturation juice filter press is called *Thin Juice Before Sulphur*. Washing is continued until a Brix spindle graduated at 160°F indicates zero in the wash water. Lime cakes from the second saturation juice are sampled and tested for sugar content in the same manner as the first saturation juice filter press cakes. An average sample of the juice from the second saturation juice filter presses should be tested for alkalinity at least once every hour.

Calculation of Sugar Lost in Lime Cakes in Per Cent to the Weight of Beets.—At the end of each period or campaign, the sugar lost in lime cakes should be computed in per cent to the weight of beets from the following formula:

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s = average sugar content in lime cakes;
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w = average weight of lime cakes in kg. per frame;

f =number of frames per press;

p = number of presses;

b =weight of beets in kg.

Sugar lost in lime cakes in per cent to beets =  $s \times \frac{w \times f \times p}{h}$ .

Composition of Lime Cakes and Their Use.—The composition of lime cakes varies within a wide limit. The foilowing is given by Andrilik as the result of analysis of lime cakes satisfactorily filtered:

Constituents in dry substance	Minimum per cent	Maximum per cent
Silica and insoluble substances	· ·53	2.99
Ferric oxide and alumina	84	4.23
Calcium oxide	. 41.31	47.13
Calcium hydroxide	14	2.49
Magnesia	· 1.71	5.13
Carbonic acid	· 26.11	33.8o
Phosphoric acid	• 1.09	2.06
Sulphuric acid	· .53	4.10
Fatty substances	05	1.29
Nitrogen	22	.36
Pentose	17	1.11
Undetermined	• 6.64	14.98

A few factories reburn lime cakes in a rotatory lime-kiln and use them for defecating the beet-juice. In some localities, lime cakes are used for fertilizing soil. A greater part of the lime cakes is lying wasted in the lime ponds of sugar factories.

Third Saturation or Sulphuring.—In most beet-sugar factories, the juice is saturated for the third time in order to further reduce the alkalinity of the juice and otherwise to improve the quality of the juice, before subjecting it to concentration. For the third saturation, sulphur-gas is used and the process is called Sulphuring. As to the effects of sulphur-gas or sulphurous acid, sugar-experts greatly differ in their opinions. The majority of eminent authorities, however, seem to agree on the following points:

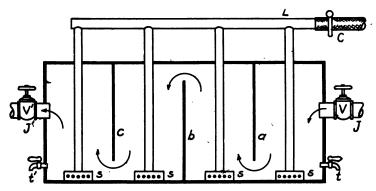


Fig. 57.—Continuous sulphuring tank.

that, besides having a power to reduce the alkalinity of juice, sulphurous acid (1) has a slight decolorizing effect, (2) precipitates lime from lime salts of organic acids that carbon dioxide fails to do, (3) facilitates the crystallization of sugar from the juice, (4) removes a considerable quantity of nitrogen compounds, and finally (5) that the sugar obtained from sulphured juice has a better keeping quality, due to the antiseptic property of sulphurous acid. The proper alkalinity of the juice after the third saturation should be about 0.010 per cent.

Apparatus for Sulphuring.—The saturation of juice with sulphur-gas is usually carried out in a continuous process. The apparatus for a continuous process is represented in Fig. 57.

It is simply a rectangular iron tank with partitions a, b, and c. There are openings at the bottom of a and c and at the top of b. L is a lead pipe which conducts sulphur-gas into the tank and is connected with wooden boxes s perforated at the bottom for discharging the gas into the juice, C, a clamp for closing the gaspipe. J and J' represent the juice-pipes, V, the entry-valve, V', the exit-valve, t, the sample-cock for the entering juice, t', that for the departing juice. Sulphur-gas is prepared by burning sulphur in an oven and is forced into the sulphuring tank by means of a steam injector or by compressed air pumped into the sulphur-oven. When the valves V, V', and the clamp C are open, the juice will flow in the direction indicated by the arrows, and while flowing, it comes in contact with sulphur-gas which is being constantly discharged from the boxes s, and becomes reduced in alkalinity and improved in quality.

Operation of Sulphur Station.—Allow the juice to flow from the trough at the second saturation juice filter presses by gravitation toward the sulphuring tank, open first the valves V and V' and then the clamp C. Take the sample first from the cock t and then from the cock t' and test their alkalinity. The flow of the juice should be so regulated that the alkalinity of the juice taken from the cock t' would test about 0.010 per cent. The faster the flow of the juice, the higher the alkalinity or vice versa. It is very important that the alkalinity of the juice is brought down to this point as this saturation is final before evaporation. When a thin juice of 0.010 per cent alkalinity is evaporated, the juice, sometimes, becomes neutral or even acid, owing to the evaporation of ammonia and to the formation of organic acids during the evaporation as is often the case with the beet-juice of poor quality. In such cases, the alkalinity of the juice should be so regulated at the sulphur-station that the juice will come out of the evaporator with from 0.02 to 0.03 per cent alkalinity. After sulphuring. the juice should be heated to boiling so that the chemical action of lime may be kept constantly active.

Filtering Sulphured Juice.—When the juice is sulphured, a considerable quantity of precipitates of calcium sulphite and some

other substances is formed. This precipitate must be removed before the juice could be subjected to evaporation. Since the quantity of precipitates resulted from sulphuring is very small, such large filtering contrivances as filter presses need not be used for this purpose. Special filters have been devised for filtering sulphured juice; they are generally called bag-filters. The one most commonly employed is that designed by Prokch. This filter is made by Breitfeld and Danek. Fig. 58 represents a cross-section of the filter. A is a cistern, hermetically closed with a cover C. In the cistern, a series of frames is suspended,

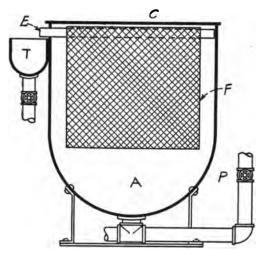


Fig. 58.-Danek filter.

one of which is represented by F. The frame is a perforated iron plate doubled like U. Each frame is attached to an exit pipe indicated by E, the open end of the frame fitting in a corresponding longitudinal slit in the pipe. Each frame is enveloped in a pillow-case-like bag. The upper borders of the bag which wrap tightly over the pipe are fastened down by means of an iron strip and screws. P represents a pipe for the entering juice, T, a trough for receiving the filtered juice. The operation of this filter is very simple; open the entry valve and allow the juice to fill the cistern A, whereupon the juice will penetrate through

the tissues of the bag, fill the frame, and flow out through the exit pipe E into the trough T. A clear juice thus obtained is called *Thin Juice After Sulphur* and pumped up to the evaporator supply tank to be drawn into the evaporators.

It is customary to determine the alkalinity, Brix degree, sugar, and purity of thin juice to see whether the operations have been properly conducted or not. The alkalinity is tested by the usual method. In determining the Brix, sugar, and purity, transfer the sample to a cylinder, cool, mix, take an accurate Brix degree, correct for temperature if necessary, determine the polariscope reading by the 110 c.c. volume method after acidifying with a few drops of acetic acid, and find the sugar and purity in Table IV.

Evaporation of the Juice.—The Brix degree of thin juice varies between 10 and 15, according to the Brix degree of the beetjuice and the amount of dilution through washing in the filter presses. Such thin juice could not be subjected directly to boiling for crystallization in a vacuum pan for the following reasons: I, the juice contains too large a quantity of water and requires too long a time to be evaporated in a vacuum pan; 2, the juice contains a considerable quantity of substances which separate from the solution when boiled down to a syrupy condition and should be removed by filtering before further concentration as they otherwise increase the ash content of white granulated sugar; 3, concentration usually increases the alkalinity of the juice which should be corrected to the proper degree, since too high alkalinity renders the crystallization of sugar difficult. It is, for these reasons, that thin juice is usually evaporated to from 55° to 60° Brix, corrected to the proper alkalinity and filtered before it is sent up to the vacuum pan for crystallization. The apparatus used for the concentration of juice is called an Evaporator.

In constructing and operating evaporators, the following points should always be kept in view: (1) The result of evaporation depends on the nature of the metal used for the heating surface. Taking the heat conductivity of silver as 1000, those of copper, brass, and iron are 736, 236, and 119 respectively. The

use of silver is out of the question owing to its expensiveness. Any of the last three may be used for the heating surface of evaporators but generally brass or copper is used. The heat conductivity of lime salts is very low, varying from 10 to 18. It is therefore very necessary that the evaporators should be emptied and cleaned every 10 or 15 days during operation.

- (2) Time and temperature being equal, the amount of water evaporated is directly proportional to the size of the heating surface of the evaporator; hence, in constructing an evaporator, the heating surface should be made as large as possible since the facilities in evaporation largely depend thereon. (3) Size of heating surface and time being equal, the amount of water evaporated is directly proportional to the temperature of the heating surface. (4) The boiling point of fluids depends on the pressure charged upon them. It is on this account that, in operating an evaporator, the vapor is condensed by means of a condenser and the air is pumped out with an air-pump in order to reduce the pressure within the evaporator so that the concentration may be conducted more economically than otherwise.
- (5) A steam of any temperature can evaporate a fluid whose boiling point is lower than the temperature of the steam. This fact is taken advantage of in concentrating juice in an evaporator consisting of many bodies called multiple effects. In multiple effects, the heating chamber of the first body is heated by the exhaust steam from the engines, the heating chamber of the second body, by the vapor developed from the juice in the first body, and likewise all the heating chambers of the succeeding bodies are heated by the vapor developed from the juice in the preceding bodies, as the juice in the succeeding bodies may be easily boiled by the temperature of the vapor developed from the juice in the preceding bodies, by diminishing the pressure on the juice in the succeeding bodies by means of an air-pump and condenser.
- (6) Steam developed by a certain temperature has the same temperature as that of the fluid from which it has been developed

so long as both remain under equal pressure; hence, in multiple effects, the temperature of the heating surface of a succeeding body would remain the same as that of the fluid in the preceding body. (7) In order to keep the boiling-point of the fluid constant, a pressure on the fluid corresponding to the boiling-point must be kept unchanged. This is very important in operating evaporators as the uniform concentration of the juice is much The evaporator man should be constantly watchful in regulating the admission of water into the condenser and the speed of the air-pump. (8) The advantages of multiple effects may be explained from the fact that I kg. of steam evaporates 0.96 kg. of water in a single effect, 1.90 kg. in double effects, 2.85 kg. in triple effects, 3.70 kg. in quadruple effects, 4.72 kg. in quintuple effects. The advantages of multiple effects may also be seen from a comparison of the coal consumed in evaporating the juice obtained from 10,000 mctr. of beets:

	metr.
with single effect	1929.7
with double effects	975.0
with triple effects	650.o
with quadruple effects	488.8
with quintuple effects	392.5

Construction of Evaporators.—There are several forms of evaporators:—one-body evaporator (single effect), two-body evaporator (double effects), three-body evaporator (triple effects), four-body evaporator (quadruple effects), and five-body evaporator (quintuple effects). Those evaporators consisting of more than one body are called multiple effects. Some evaporators are vertical, some horizontal. In this work, just vertical effects will be briefly described. Fig quadruple evaporator consisting represents a vertical of four bodies indicated by I, II, III, IV. Each body consists of two sections of large cast-iron cylinders. The lower section H is the heating chamber; the upper section V, the vapor chamber. The heating chamber is pierced through by numerous brass tubes which serve as a heating surface as well as for the passage of juice. H' represents the valve which admits exhaust steam from

the engines and pumps into the heating chamber of the first body; J, the juice-pipe which supplies the evaporator with juice from a supply tank; J', the juice-valve; A, a water-pipe; W, a water-valve; s, a space in the heating chamber filled with steam; j, a passage for juice; j', a pipe leading the evaporated juice to the thick juice pump; P', an outlet valve; K, a condenser which is simply a tall iron cylinder provided with baffle plates; W', the valve which admits water into the condenser; A', a pipe which takes water away from the condenser; P, a pipe which carries air from the condenser to the air-pump; t, a pipe which car-

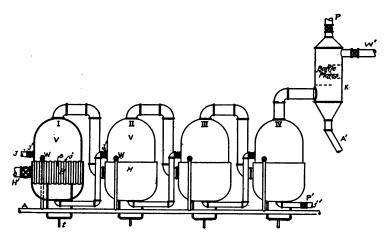


Fig. 59.—Quadruple effects evaporator. (Horizontal type).

ries water condensed in the heating chamber to traps. Each body of evaporator is provided with a vacuum-gauge and a thermometer for regulating the pressure and temperature. Each body is also provided with a glass gauge to indicate the height of the juice within. It is also provided with grease cups so that grease may be introduced into the juice chamber in case the juice foams too violently. The heating chambers of the second, third, and fourth bodies are communicated with the uppermost parts of the vapor chambers by means of small pipes. These pipes are called ammonia pipes, as they carry away ammonia gas collected at the upper part of the heating chambers.

These pipes also facilitate the circulation of the vapor. Some ammonia pipes are connected directly with the condenser.

When the evaporator is in operation, all juice-valves are open, all bodies are filled with juice to about three-quarters the height of the heating chamber, exhaust steam is admitted into the heating chamber of the first body, the air-pump is running, and water is admitted into the condenser. Under these conditions, the temperature and pressure in each succeeding body are lower than those of the preceding body, being highest in the first body and lowest in the fourth. The vapor evolved from the first body will pass over into the heating chamber of the second body and boil the juice owing to a diminished pressure therein caused by the air-pump and condenser; the vapor evolved from the second body will pass over into the heating chamber of the third body and boil the juice, owing to a still further diminished pressure therein, and so on.

Operation of Evaporator.—Start the air-pump, admit a little water into the condenser, and open the juice-valve of the first body and the exhaust steam valve H, whereupon the juice will enter the first body and boil therein. The vapor evolved from the juice will pass over into the heating chamber of the second body, serving as the exhaust steam did in the heating chamber of the first body. When the juice covers the top of the heating chamber of the first body, which may be seen through eye-glasses, close the juice-valve of the first body so that the first body may not be filled too full and open the juice-valves of the second, third, and fourth bodies successively, whereupon the juice will be introduced into those bodies one after another, while the vapor evolved from the juice in the second and third bodies will pass. over into the heating chambers of the third and fourth bodies. When the juice in the first body is reduced to about three-quarters the height of the heating chamber which may be seen in the glass gauge, open the juice-valve of the first body again. The admission of the juice into the first body should be so regulated that it will remain at that height nearly constantly. The juice-valves of the second, third, and fourth bodies should also be so regulated that the juice will remain in those bodies at nearly the same height as that in the first. As the juice advances, admit more water into the condenser. When the evaporator is running normally, the admission of water into the condenser should be so regulated that the juice in the fourth body will boil at 60°C (140°F) and 23 inches vacuum; the admission of exhaust steam into the heating chamber of the first body should be so regulated that the juice in the first body would boil at 104°C (220°F) and 2.3 pounds pressure. The juice in the fourth body should be tested by means of a Brix spindle. As soon as the spindle indicates from 55° to 60°, open the valve P' and draw the juice by pumping. It is a common practice to so regulate the flow of the juice that the Brix degree of the juice in the fourth body remains at from 55° to 60° and to keep the pump going almost without intermission.

Chemical Examination of Thick Juice.—The juice thus concentrated is called Thick Juice. It is the usual practice to subject the juice to another saturation with sulphur-gas. The juice, previous to this saturation, is called Thick Juice before Sulphur, and following the saturation, Thick Juice after Sulphur. Thick juice before sulphur should be examined for alkalinity, Brix, and purity. It is important to know the alkalinity of the thick juice before sulphur in order that the alkalinity of the thin juice may be regulated at the sulphur station. A sample of the juice for alkalinity test should be taken directly from the fourth body once every hour and be tested in the chemical laboratory by the usual method. Examination of the juice for Brix is also important in order to regulate the operation of evaporators and also to keep the sugar-house management well informed as to the efficiency of the evaporators. A sample should be drawn from the fourth body every 15 minutes, be saved in a sample bottle for a specified interval of time, usually two hours. At the end of the period specified, take the sample saved to the chemical laboratory, mix, transfer to a cylinder, cool down to about normal temperature, mix again, Brix, correct if necessary, and record as Brix of thick juice before sulphur.

The most important chemical examination of thick juice at this stage is that of the purity thereof, for it is very necessary that sugar-experts should be constantly informed of the relative purity of the juice after and before evaporation and also the relative purity of thick juice before and after sulphur. Dilute a small portion of the sample saved for the Brix test with pure water in a cylinder to about 20° Brix, take accurate Brix degree, determine the polariscope reading by the 110 c.c. volume method in the usual manner and find the purity in Table IV.

Emptying and Cleaning Evaporator.—As the juice concentrates, salts of lime become less soluble in the juice and separate as precipitates, which partly deposit on the heating surface and partly remain suspended in the juice. In the course of from 10 to 15 days, the deposit of lime salts on the heating surface would become so thick that it would greatly diminish the efficiency of the evaporator, owing to the poor heat conductivity of lime salts. Then it is necessary to empty the whole evaporator and When the last portion of the juice to be clean thoroughly. evaporated is drawn into the first body, distribute the juice nearly equal in all the bodies, close all the juice-valves, and then boil down in each body separately until the usual Brix degree of the juice is nearly reached. Boiling may be facilitated by raising the temperature of the first body to about 115°C (239°F) and the pressure to 10 pounds. When a desired Brix of the juice is attained, open all the juice-valves excepting that of the first body, draw the juice from the fourth body by pumping and open the vacuum-breaking-valve of the first body as soon as the vacuum gauge indicates the existence of any vacuum therein. When the first body is empty, which may be found by watching the vacuum gauge of the second body for a sudden drop of the vacuum due to the admission of air through the juice-valve, close the juice-valve, open the vacuum-breaking-valve of the second body and fill the first body with water until 2 or 3 inches of water cover the top of the heating chamber. When the vacuum gauge of the third body shows a sudden drop of the vacuum.

indicating that the second body is empty, close the juice-valve, open the vacuum-breaking-valve of the third body and fill the second body with water. As soon as the vacuum gauge of the fourth body shows a sudden drop of vacuum, close the juicevalve of the fourth body and the condenser water-valve W', stop the air-pump, open the vacuum-breaking-valve of the fourth body and then fill the third body with water. When the thick juice pump no longer sends up juice to the blow-up supply tank, close the outlet valve P', fill the fourth body with water. When a sufficient vacuum is created in the first body by the action of cold water, introduce about 125 pounds of caustic soda already dissolved in water through a small tube which opens into the juice chamber just below the heating chamber, then admit exhaust steam into the heating chamber of the first body. Now start the air-pump and admit water into the condenser. As soon as a sufficient vacuum is created, introduce into the second, third, and fourth bodies, respectively, about 125, 200, and 300 pounds of caustic soda already dissolved in water and continue boiling for 4 hours. No juice-valve should be opened during clean-At the end of the time specified for boiling, close the exhaust steam-valve H' and the condenser water-valve W', stop the air-pump, open the vacuum-breaking-valves, and empty each body separately by opening the sewer-valve provided at the bottom of each body. When they are completely empty, fill them with water again until 2 or 3 inches of water cover the top of the heating chamber, close the vacuum-breaking-valves, start the air-pump and condenser, introduce into the first, second, third, and fourth bodies respectively, I, I, 2, and 3 carboys of muriatic acid in the same manner as caustic soda was introduced and boil 4 hours again. After the boiling is completed, empty them the same as before, fill them with water again in order to wash out the acid, empty, free the tubes from the deposits of lime salts by scraping with suitable tools, rinse out all the dirt, and close all the valves. Now the evaporator is ready for use again.

Saturation of Thick Juice.—As was stated previously, the alkalinity of thin juice, having a Brix degree from 10 to 12, is about 0.01 per cent. The alkalinity of such juice, when concentrated to Brix from 55° to 60° would be raised to about 0.05 per cent, but owing to the combination of lime with some organic acids, it is usually reduced to from 0.02 to 0.03 per cent. It is, however, still too high to be sent up to the vacuum pan. It is a common practice to reduce the alkalinity of thick juice to from 0.005 to 0.010 per cent with sulphur-gas before concentrating it to crystallization. The apparatus used for this purpose is called a Blow-up. It is a round iron tank provided with a perforated steam coil for heating and a perforated wooden box into which a lead sulphur-gas pipe opens.

In operating this station, fill the tank with the thick juice from the evaporators about three-quarters full, heat it to boiling and test the juice for alkalinity by the usual method. Should it exceed the proper point, saturate it with sulphur-gas until a desired alkalinity is attained. In case it is lower than 0.005 per cent, or neutral or even acid, it should not be saturated. If acid, it would be of some advantage to add enough lime to bring up the alkalinity to about 0.005 per cent. When saturation is completed, open the outlet valve, allow the juice to flow into a Danek filter through which the juice will come out free of suspended matter. It is very necessary to filter thick juice after evaporation and saturation, as it contains precipitates of lime salts suspended which would increase the impurities in granulated sugar if not filtered off. Clear thick juice thus obtained is pumped up to the vacuum pan supply tank ready to be boiled down to crystallization.

Chemical Examination of Thick Juice after Sulphur.—It is a common practice to examine thick juice after sulphur for alkalinity, Brix, and purity in the chemical laboratory in order to see whether or not saturation has been conducted properly and any improvements are made thereby. The methods for testing the alkalinity, Brix, and purity of thick juice after sulphur are the same as those applied for thick juice before sulphur. An average sample should be taken from the Danek filters every 2 hours.

Principle of Sugar-Boiling.—Thick juice thus obtained con-

tains, besides sugar, compounds called non-sugars, varying from 10 to 20 per cent to the weight of sugar. Non-sugars are more or less soluble than or equally as soluble as sugar in water. Those which are less readily soluble than or equally as soluble as sugar crystallize out with sugar from a solution on concentration. Those which are more readily soluble than sugar remain in solution as long as enough water is present and go over with the syrup. The difference in solubility makes it possible to separate sugar from non-sugars and obtain sugar in a purer state. The aim of sugar-boiling is to accomplish crystallizing sugar and holding back non-sugars in solution. The presence of non-sugars always interferes with the crystallization of sugar when the solution capable of crystallizing is a homogeneous mixture of molecules of sugar and non-sugars. The formation and growth of sugar crystals depend on the attraction and juxtaposition of molecules of sugar on the molecules of sugar already crystallized; it is evident, then, that the molecules of non-sugars interposing between the molecules of sugar will offer obstacles to the migration and reunion of sugar molecules; and consequently when a large quantity of non-sugars is present, sugar-crystals remain small and dull. The presence of uncrystallizable nonsugars exercises a definite influence on the quality of the syrup. A solution of pure sugar, even in concentrated forms, retains a certain degree of fluidity and consequently may be comparatively easily separated from crystals; on the other hand, a syrup containing a large quantity of uncrystallizable substances is thick and sticky, even assumes a tenacious quality and makes its separation from crystals extraordinarily difficult or even impossible when the concentration is carried out too far. An increase of nonsugars in a sugar solution will offer correspondingly greater hindrance to the crystallization of sugar and at a certain point no more sugar will crystallize out, the resulting product being uncrystallizable molasses.

From what has been said, it can be seen that sugar may be boiled to crystallization by different methods according to the quality of the juice. There are two common methods for boiling

sugar:—Blank-Boiling and Boiling-on-Grains. In blank-boiling, a sugar solution is boiled down to a certain density and crystallization is effected by gradual cooling. In boiling-on-grains, crystals are allowed to form and grow during boiling. In the process of blank-boiling, one should aim to boil the juice to a supersaturation so that there will be but little or no crystals and a large part or nearly all remain as syrup and to effect crystallization largely by cooling. Under these conditions, the liquid will remain so sufficiently fluid that the molecules of sugar will be less hindered in their movements and separate in large crystals. Should the concentration be carried very much further, the crystals formed would be very small and the separation of syrup therefrom would be very difficult.

On the other hand, when a juice contains a small quantity of non-sugars, it may be boiled to a greater concentration, and crystallization may be allowed to take place while boiling, as the non-sugars offer but a little hindrance and the crystals will grow large, well built and separate from the adhering syrups very readily. Boiling-on-grains is usually employed when sugar is to be boiled from beet-juice, wash syrup, melted sugar or other material of high purity. Either white sugar or raw sugar may be boiled by the method of boiling-on-grains. When a pan of juice is boiled for producing white sugar, it is called a *Straight Pan*; when boiled to produce raw sugar, a *Raw Pan*. Syrups separated from the fillmasses of straight pans are too rich in non-sugars for boiling-on-grains. Such material is boiled down to a supersaturation by blank-boiling and crystallization is effected by cooling in a contrivance called a crystallizer.

Apparatus for Boiling Sugar.—Formerly, sugar was boiled in open pans over a direct flame or by heating with steam-coils. Sugar crystals produced by boiling in such a pan acquire a brown color, owing to the decomposition of sugar by high temperature. The fuel consumption also is much greater in the open pan process than in the other modern system. For these reasons, the open pan has been abandoned in most sugar factories. A modern apparatus used for boiling sugar is called a

Vacuum Pan. Strictly speaking, sugar is not boiled in a vacuum pan but in a pan in which pressure is diminished by means of an air-pump and a condenser. Under a diminished pressure, a sugar

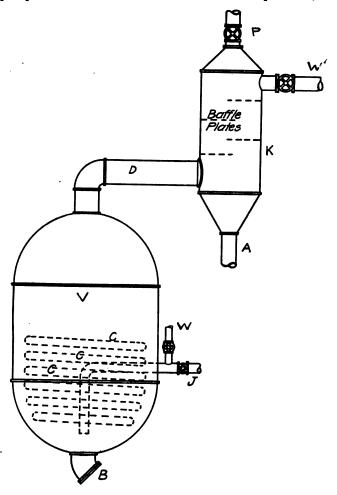


Fig. 60.-Vacuum pan.

solution may be boiled at a lower temperature and the decomposition of sugar may be minimized.

Fig. 60 represents an elevation view of a vacuum pan. V

represents the body of the pan; c, the copper steam-coil for heating; J, the juice-valve; W, the water-valve for admitting water into the pan; B, the outlet cover; D, the pipe for conducting vapor into the condenser; K, the condenser; W', the water-valve for admitting water into the condenser; P, the pipe leading air from the condenser to the air-pump; A, the pipe for carrying water away from the condenser. Fig. 61 represents a plan view of an individual steam-coil. s represents the valve for admitting steam into the coil; o, the outlet valve for steam. All vacuum pans

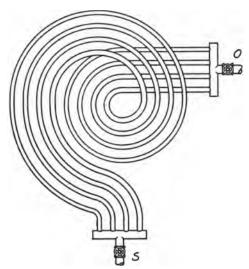


Fig. 61.—Stem coils for vacuum pan.

are provided with eye-glasses, a vacuum-breaking-valve, a thermometer, and a vacuum gauge.

Regulations Generally Adopted in Sugar-Boiling.—(1) It is important to have the juice or syrups to be used for boiling sugar examined for purity before drawing into the pan so that the sugar-boiler may be informed of what quality of juice he is to deal with. They are tested in the same manner as thick juice.

(2) The amount of juice or syrup to be drawn into the vacuum pan for starting varies according to total quantity to be had

for boiling one complete pan. Generally about 40 per cent of the

total is drawn in for the first charge. When there is enough juice to boil a full pan in a vacuum pan provided with six steam-coils, the juice is drawn into the pan until it just reaches the fifth coil for the first charge, boiled down to the fourth coil and kept there by drawing juice in from time to time until it begins to grain.

- (3) As to temperature and vacuum during boiling, it is very necessary to keep within the limits established by long experience. Stammer recommends it as best to boil at a temperature between 75° and 62°C (167°-144°F) in the juice and under a vacuum between 24 and 26 inches. Stohman recommends to start boiling at 60°C (140°F) in the vapor and 610 m.m. vacuum (24.4 inches) and to grain at 50°C (122°F) in the vapor and 668 m.m. vacuum (26.3 inches). The latter author also recommends to gradually decrease the temperature toward the end of boiling as there is danger of forming fine crystals when hot fillmass is run out of the pan and cooled off.
- (4) It is very important to have practical methods for testing the concentration of syrup as Brix spindles could not be used for a liquid having a Brix higher than 80°, owing to rapid formation of crystals on cooling and to too high viscosity. There are three practical methods in common use for testing high concentrations of juice:—string proof, bubble proof, and water proof
- (a) String Proof.—Place a small portion of the boiling mass obtained from the pan by means of a proof stick on the thumb, rub it with the index finger of the same hand and separate the fingers in a vertical direction; then three cases may be observed: 1, the mass shows no string; 2, the mass produces a string which does not break even when the fingers are separated as wide as possible; 3, it produces a string which breaks when it reaches a certain length. In the first case, the mass is not sufficiently concentrated; in the second case, the mass is too concentrated; in the last case, a proper concentration may be judged from the length and thickness of the string. The manner of breaking also indicates the degree of concentration.
  - (b) Bubble Proof.—Dip a spoon in the boiling mass, shake

off the excess of juice and blow quickly against the spoon. There will be no bubbles formed in the mass covering the opening of the spoon when the juice is sufficiently concentrated. The size, gloss, lightness, and firmness of the bubbles indicate to the accustomed eyes of a sugar-boiler exactly the degree of concentration of the syrup. The smaller, glossier, lighter, and firmer the bubbles are, the more concentrated is the juice.

- (c) Water Proof.—Drop a small portion of the boiling mass in cold water. As soon as the drop forms a ball which does not cling to a finger and flattens itself by its own weight, boiling should be terminated.
- (5) The amount of fresh juice to be added to the mass in the pan should be carefully proportioned. It is a common practice to add in the proportion of 2 liters of fresh juice to 100 liters of the mass in the pan. With such a proportion, the mass will be diluted very slightly and the excess of water will be evaporated in a very short time. With a larger quantity of fresh juice, there is danger that the mass will be rendered so thin as to dissolve the crystals already formed.
- (6) When it is desired to obtain coarse grains, it should be boiled as quietly as possible, minimizing the motion of the mass, for under this condition, molecules of sugar which are passing from a fluid to solid condition prefer to deposit themselves on the surface of the crystals already formed rather than to form new crystals, thus effecting the growth of the crystals. On the other hand, if it is desired to obtain fine grains, lively boiling should be adopted as the strong motion of the mass favors the development of new individual crystals. The sizes of grains may also be controlled by regulating the amount of juice for each addition and the frequency of the addition of juice. When a small quantity of juice is added at short intervals, the crystals would be prevented from marked growth and fine grains be formed. On the other hand, if each succeeding addition was increased in quantity and delayed a little longer, the crystals make a considerable growth during the delay. The increase of the amount of juice should be carefully proportioned as a large excess would dissolve the crystals that are to be preserved.

- (7) As the mass concentrates, naturally new fine crystals appear which should be carefully dissolved by a fresh addition of juice. Every soluble substance dissolves so much the quicker, the greater the points of contact it offers to a dissolving liquid. The surface of finely divided particles of a certain quantity of a substance is much greater than that of the same weight of the substance consisting of larger particles. The number of points of contact is always proportional to the size of the surface; it is evident, then, that, under similar circumstances, smaller crystals would dissolve much quicker than larger ones. The small crystals dissolved by a fresh addition of juice serves to hasten saturation of the freshly added juice. On further concentration, this dissolved sugar will deposit on the surface of the larger crystals effecting a corresponding growth.
- (8) The time for the termination of boiling may be recognized from the quality of the fillmass. There should be a proper amount of crystals in the fillmass, that is, the fillmass should contain so much crystals and so little syrup that the proof will not flow away when placed on a solid body. No definite limit, however, may be laid down in this respect as a concentration of fillmass must be varied according to the quality of the juice and the sizes of the crystals. When the juice is pure and the crystals are large, the mass may be boiled down until a very little syrup is left as it would still remain fluid enough to freely separate from the crystals. In case the juice is less pure and the crystals are small, boiling should be terminated a little earlier, as the syrups of low purity cling to crystals more tenaciously, and fine crystals offer more points of contact, and consequently, the separation of crystals from syrups would be more difficult.
- (9) Sugar-boiling may not always be conducted as smoothly as desired. The usual difficulties sugar-boilers have to contend with are foaming and slow boiling. Foaming is caused by slimy conditions of the juice and takes place at the beginning of boiling. It is usually remedied by introducing a spray of steam or a small quantity of tallow. When the content of the pan is foaming violently, the interior of the pan should be constantly

watched so that the steam-valve shall be closed and the air-valve or vacuum-breaking-valve be opened at the moment the juice begins to rise.

Slow boiling is the worst of all difficulties. Sometimes when the syrup reaches a certain concentration, boiling suddenly stops without reducing the temperature or vacuum, and the mass remains very quiet. Increase of heat or vacuum is unable to bring the mass to boiling again. The mass gradually becomes brown owing to the caramelization of the sugar. Slow boiling is generally attributed to two causes: I, too strong alkalinity; 2, the presence of an abnormal quantity of lime combined with organic compounds. Too strong alkalinity is caused by defective treatment of the thick juice during saturation. It may be remedied by neutralizing with calculated amount of sulphuric, muriatic, or phosphoric acid after determining the alkalinity. presence of an abnormal quantity of organic lime salts is usually attributed to defective operations in diffusion, liming, and saturation, but often is the consequence of unfavorable composition of beets caused by harvesting unripe beets or by poor conservation. Slow boiling caused by a presence of organic lime salts may usually be remedied instantly by introducing a solution of sodium sulphite after determining the lime by means of This remedy, however, is merely a make-shift as soap solution. calcium sulphite remains insoluble in fillmass and increases the ash content of granulated sugar, hence it would be much better to remove the lime from thick juice at a proper time so that a slow boiling would not generally result.

Boiling Straight Pan.—See first that all the valves, outlet cover, man-hole and vacuum-breaking-hole are closed, and also estimate the juice or syrups on hand or that could be secured in the immediate future, as it is necessary to anticipate and vary the amount of the juice or syrup for the first charge. Set the airpump in action and admit water into the condenser in order to create a vacuum in the pan. When a sufficient vacuum is created in the pan, draw juice or syrup into the pan up to the fifth coil, admit steam into the lowest four coils covered with juice, where-

upon the juice begins to boil, regulate the water in the condenser so that the vacuum would remain at about 24 inches and the temperature of the juice, at about 75°C (167°F). When the juice is boiled down to the fourth coil, draw some more juice in and keep the fourth coil just covered with the juice until graining commences. As soon as the boiling mass begins to show a string proof and a few crystals appear in the mass, which may be recognized in a proof taken out of the pan by means of a proof stick and placed on a glass plate, give the boiling mass in the pan a lively motion by opening the juice-valve for a few seconds. Lively motion helps to hasten the formation of grains. This should be repeated until enough grains are obtained. In the course of a few minutes, the juice will become more concentrated and the growth of crystals will appear slow, then add some more juice in the proportion of 2 liters of fresh juice to 100 liters of the syrup contained in the pan. Henceforth, a proof should be taken every few minutes and as soon as the syrups between grains lose ready fluidness, and the grains become closely crowded, fresh juice should be added. The sugar-boiler must exercise the utmost care to avoid supersaturation which may be readily recognized by a milky appearance of the syrup in a proof placed on a glass plate. Syrup should always appear perfectly clear. saturation may be remedied by promptly adding fresh juice and by slacking water into the condenser to slightly raise the temperature until the syrup becomes perfectly clear. By continuing the concentration of the syrup and the addition of fresh juice, the volume of the mass will gradually increase and cover the remainder of the steam-coils one after another. Steam must be admitted into each coil as soon as it is covered with the syrup. When the pan is sufficiently filled, boiling should be continued without adding fresh juice. As the concentration of syrups remaining in the spaces between the sugar crystals continues, sugar molecules, if boiled properly, deposit themselves on the surface of the crystals already present. After the addition of fresh juice is discontinued, boiling should be conducted very slowly so that the formation of new fine grains may be minimized. When the mass in the pan becomes so destitute of fluidness that a proof placed



on a glass plate will not flow away, boiling is complete. The product is called *Fillmass* (German) or *Massecuite* (French). When the boiling is completed, close all the steam-coil valves and the condenser water-valve, stop the air-pump, open the vacuum-breaking-hole and then the outlet cover and allow the fillmass to flow into a reservoir called the *Mixer*.

Chemical Examination of Fillmass.—It is a common practice to examine fillmass for purity so that the sugar-house management may be informed of whether or not the process has been conducted properly. Take a sample while the fillmass is flowing from the pan into the mixer, dissolve completely a small portion of the sample in pure water and make a solution of about 20° Brix. Now take accurate Brix, correct for temperature if necessary, determine the polariscope reading by the 110 c.c. volume method and find the purity in Table IV. When of a determination of the Brix, sugar, and purity of undiluted fillmass is desired, weigh out 3 normal weight of the sample, dissolve in pure water, and make it up to 300 c.c. This is normal solution. Now take the Brix, determine the polariscope reading by the 110 c.c. volume method, and find the purity in Table IV. The sugar in the undiluted sample of fillmass would be polariscope reading + polariscope reading since 100 c.c. of the normal solution are diluted to 110 c.c. As stated previously, purity is obtained by dividing sugar by Brix and multiplying by 100, that is, purity =  $\frac{\text{sugar} \times 100}{\text{Brix}}$  or  $P = \frac{S \times 100}{B}$ ; hence, Brix may be obtained by dividing sugar by purity and multiplying by 100, that is,  $Brix = \frac{Sugar \times 100}{purity}$  or  $B = \frac{S \times 100}{P}$ . apparent then that the Brix of undiluted fillmass may be obtained by dividing the polariscope reading + its one-tenth by purity and multiplying by 100. In Table IV, Brix degrees for undiluted substances are given in the horizontal line directly

above Brix degrees found for normal solutions.

## EXAMPLE.

Brix degree of normal solution	22. I
Polariscope reading by 110 c.c. volume method	72.2
Purity	85.5
Sugar in undiluted fillmass $\cdots 72.2 + 7.2 =$	79.4
Brix degree of undiluted fillmass $\frac{79.4}{85.5} \times 100 = 9$	92.8.

The last value is found directly over the Brix degree of normal solution 22.1 in Table IV.

Disposition of Fillmass.—Fillmass thus obtained is subjected to centrifugation in order to separate the sugar from the syrups. The sugar is dried, weighed, bagged, and sent to market as granulated sugar. The separation of sugar from the mother syrup and drying and bagging the sugar are purely mechanical, therefore, in this work, only a brief description will be given on these subjects.

Separation of Sugar from Syrups.—Separation of sugar from syrup is accomplished by a contrivance called a Centrifugal Machine. The machine is simply a short, wide brass cylinder with perforated walls lined with a fine brass screen in order to keep the sugar from passing out of the cylinder. It is suspended by means of a steel rod from the bottom of the mixer and enclosed in a cast-iron shell. It is usually driven by means of a belt at a speed of 1200 revolutions per minute. In operating this machine, it is filled with fillmass about one-third full and set in motion, whereupon the centrifugal force will drive the mass against the walls of the machine and force the syrup out through the pores in the screen, while the sugar crystals remain clinging to the inner surface of the screen. The syrup thus spun out is called Green Syrup. The syrup driven against the walls of the shell collects at the bottom thereof and then flows through a spout into a trough which conducts the syrup to the green syrup tank. The last trace of syrups adhering to the crystals is washed off by sprinkling water while the machine is still in motion. The syrup produced by washing is called Wash Syrup. The syrup spout is shifted to a wash syrup trough before washing commences so that the wash syrup may be stored in a separate

tank. Wash syrup is diluted to about 60° Brix, filtered, and used for boiling white sugar mixed with thick juice.

Examination of Green and Wash Syrups for Purity.—It is a common practice to examine green and wash syrups for purity in order to control the process of sugar boiling as well as for determining their disposition. A sample of green syrup is taken directly from the green syrup spout. In order to obtain an average sample, it is necessary to catch the syrup several times at intervals of a few minutes, holding each time a sample pail under the spout for a few seconds. It should be so arranged that the sampling would cover the whole period of spinning one charge of fillmass. The wash syrup sample is also taken in the same manner. The methods for the determination of the purity of green and wash syrups are the same as those employed for fillmass.

Transporting, Drying, and Bagging of Sugar.—When the washing is completed, the sugar is dumped into a trough situated directly below the centrifugal machines. The sugar in the trough is forwarded by means of a scroll to one end of the trough where it is transferred to an elevator, small metallic buckets fastened on a belt, and then lifted up to a sugar storage box which is located on the upper floor. The storage box is provided with a hopper which directs the sugar into a sugar drying machine called A granulator is simply a large, long cylinder a Granulator. made of sheet-iron supported on a series of roller wheels, inclining toward the outlet end and rotated by means of a wheel gear. The interior of the granulator is heated by admitting steam into a pipe which passes through the center of the cylinder throughout the entire length. At the outlet end, a brass screen is attached to the cylinder so that any lumps of sugar may be kept from going into the bagging room. In operating, the granulator is set in motion and sugar is admitted thereinto from the storage box through a hopper at such a speed that the out-going sugar is perfectly dry. The damp sugar admitted into the granulator, while moving on toward the outlet, will come in contact with hot air, be dried, screened, and pass into a hopper which directs the dried sugar to the bagging room, where the sugar is weighed and bagged. Sugar bags are double, the inner bag being made of cotton and the outside bag, of burlap. They are all numbered and marked with the name of the refinery.

Blank Pan.—The syrup separated from a straight pan fillmass by means of the centrifugal machine is a saturated solution in cold which is much richer in non-sugar than thick juice. Sugar in such a solution may be brought to crystallization by further concentration. The method of boiling-on-grains, however, could not be carried out with such syrup, owing to the presence of a large quantity of non-sugars which prevent the migration of sugar molecules by interposing between them. Such syrup should be subjected to blank-boiling and crystallization be effected by a slow cooling.

Prior to boiling, warm the syrup in storage tanks in order to reduce the tenaciousness, which makes the drawing into the pan difficult. Start the air-pump, draw so much syrup as to cover all the steam-coils, admit steam into the coils, and set the condenser in action. As soon as boiling begins, so regulate the water into the condenser that a vacuum in the pan will remain at about 610 m.m. (24 inches) and keep the juice-valve open so wide as to introduce continually enough juice into the pan to The syrup will boil lively compensate the water evaporated. while thin, but as the concentration advances, it will boil more quietly. The concentration may be constantly watched by the string proof, and the water proof may be used for a final test. It is a common practice to examine blank pan fillmass for Brix, sugar, and purity. Analysis of this product is made in the same manner as that of straight pan fillmass.

When the boiling is completed, close the steam-valves, turn off the water from the condenser, stop the air-pump, and open the vacuum-breaking-hole and the outlet cover, whereupon the fillmass will flow into a crystallizer through the trough. A crystallizer is simply a large cylinder made of sheet-iron, provided with an agitator and a water-jacket. In operating this machine, fill with fillmass, close the cover, admit water into the

water-jacket in order to gradually reduce the temperature of the mass and then set the agitator in motion so that the temperature will be kept uniform throughout the mass and the crystals once formed will be in constant access to the molecules of sugar in solution ready to supply for their growth. The agitator may be kept in motion for about one-half hour at intervais of 2 or 3 hours. When the temperature of the mass is brought down to about 46°C (115°F) and the syrup becomes clear and soft, crystallization may be regarded as complete. Then the mass is emptied into a mixer. Blank pan fillmass is also subjected to centrifugation in order to separate sugar from the syrup but a smaller quantity of water is used for washing. Wash syrup from the blank pan fillmass is mixed with green syrup from a straight pan and used for boiling a blank pan again. Sugar thus obtained is called Yellow, Brown, or Raw Sugar. Syrup from a blank pan fillmass is called Molasses. Raw sugar is melted with condensed water or thin juice to about 60° Brix, filtered, and used for boiling a straight pan.

Special Regulation Adopted for Keeping the Purity of Molasses Low.—It has been found by experience that the higher the purity of molasses, the greater is the loss of sugar, or vice versa; hence, the loss of sugar may be minimized by keeping the purity of molasses as low as possible. The regulations to keep the purity of molasses low should begin with the first pan massecuite. Other things being equal, massecuites of high purity produce green syrups of high purity. When the purity of the first pan massecuite is high, the purity of the first green syrup is invariably high. When a massecuite is produced by boiling a high green syrup, the purity of the molasses separated from such a massecuite would also be high. Hence, it is advisable, in boiling a first pan, to use green syrup toward the end of boiling so that some sugar may be taken away from the green syrup by the sugar crystals already present, thus lowering the purity of the syrup. The excessive use of green syrup, however, should be avoided since it will very likely give a yellow cast to the sugar crystals. The addition of green syrup should be so proportioned as to reduce the purity of the massecuite to from 84 to 87 per cent and that of the green syrup to from 70 to 74 per cent. It is also advisable to reduce the purity of the raw pan massecuite when it is abnormally high by adding some molasses of high purity. The proportion of molasses should be such as to reduce the purity of the massecuite to from 70 to 74 per cent and that of the molasses separated therefrom to less than 60 per cent.

Chemical Examination of Yellow Sugar and Syrup.—Analysis of yellow sugar and syrup is very necessary as it gives important bearings in controlling sugar boiling. Yellow sugar is usually examined for sugar and purity. Weigh out 3 normal weight in a sugar-dish, add a little water, dissolve completely in the dish, transfer to a 300 c.c. flask, make up to the graduation mark with water, mix thoroughly, pour into a glass cylinder, take the Brix, determine the polariscope reading by the 110 c.c. volume method, and find the purity in Table IV. The polariscope reading + one-tenth the polariscope reading will give the sugar. Molasses should be examined just for purity. Dilute a small portion of molasses to be examined to about 17° Brix, take accurate Brix, determine the polariscope reading by the 110 c.c. volume method, and find the purity in Table IV.

Disposition of Molasses.—Sugar in molasses could not be separated by the process of crystallization, owing to the presence of a large quantity of non-sugars as may be seen from the following analysis:

Constituents	Per cent.
Sugar	· 50.0
Non-sugar ·····	. 30.0
Water	. 20.0
•	
•	100.0

There are numerous methods for the separation of sugar from molasses. In this work, only two methods—Osmose process and Steffen's process—will be briefly described, as they are most commonly adopted in this country.

Osmose Process.—The principle of osmose is identical with that of membrane diffusion. As stated under PRINCIPLE OF

DIFFUSION, various substances have different powers of diffusing through a membrane. Many inorganic salts, such as chloride and nitrate of potassium and corresponding salts of sodium, are more readily diffusible through a membrane than sugar. This fact is taken advantage of in partially removing the non-sugars in molasses. In applying this principle, streams of molasses and water are brought in contact, not directly, but separated by a parchment paper, whereupon the water diffuses through the paper into the molasses, while the more readily dif-

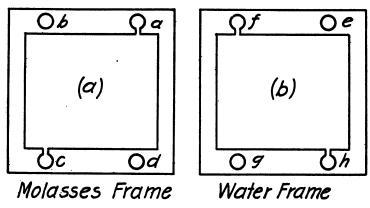


Fig. 62.—Osmose frames.

fusible salts in the syrup pass out into the water. This process is called Osmose.

The apparatus used for this purpose consists of a number of frames pressed together, all consecutive frames being separated by a sheet of parchment paper. The frames are of two kind one is a syrup frame, the other, a water frame. In Fig. 62, (a) represents the molasses frame, (b), the water frame. The frames are provided with openings a, b, c, d, e, f, g, h, for passages of syrup and water. a, c, f, and h are open into the interior of the frames. When in use, these frames are placed alternately, all two consecutive frames separated by a sheet of parchment paper and the whole pressed tight by means of a screw. Under these conditions, when a syrup is admitted into c, it will fill the molasses frame and flow out through a; as e has no opening

into the interior, the syrup will flow, without entering the water frame, into the next molasses frame through a; as g is not open into the interior, the syrup will pass from the second molasses frame to the third, and continue its course in like manner to the end of the apparatus. On the other hand, when water is admitted into h, it will fill the water frame and flow out through f; as b has no opening into the interior, the water will pass by the molasses frame and fill the next water frame; as d is not open into the interior, the water will pass from the second water frame to the third, and continue its course in like manner to the end of the apparatus, without coming in direct While thus flowing, an interchange of contact with molasses. salts and water through the parchment paper is effected, diluting the molasses and at the same time improving its purity considerably. A small quantity of sugar also diffuses through the paper during the operation. In starting the operation of this apparatus, syrup and water are admitted from the same end so that there will be an equal pressure on the paper from both sides, but as soon as the apparatus is full, they are admitted from the opposite direction so that the syrup, as it advances, would come in contact with water containing less salts and finally with fresh water.

Osmotic action takes place best in hot water; on this account, the water is usually heated up to 90°C (194°F) and the molasses up to 80°C (176°F) before they are admitted into the apparatus. The proportion of water and molasses could not be definitely fixed as it should be varied according to the quality of molasses. Under normal conditions, however, it is so regulated, that Brix spindles graduated at 75°C (167°F) would indicate from 35° to 40° in the out-going molasses and 3° in the out-going water. The proportion of 5 parts of water to 1 part of molasses by volume would usually bring about these results. Out-going molasses is called Osmose Juice, out-going water, Osmose Waste Water. Osmose juice thus obtained is concentrated in an evaporator to about 60° Brix and used for boiling blank or raw pan. The following are the average results of osmose process of a well conducted sugar-house.

mo	itering classes r cent.	Out-going molasses per cent.	Out-going water per cent.
Brix degree	70.10	33.71	2.96
Sugar	13.77	23.86	.82
Purity	2.43	70.78	27.70

Chemical Analysis of Osmose Products.—Entering molasses is usually examined for Brix, sugar, and purity in order to compare them with those of osmosed molasses. Molasses may be analyzed in the same manner as fillmass. The alkalinity of the molasses is also examined before entering this process.

Samples of osmose juice and waste water should be taken frequently and the average sample should be examined for Brix, sugar, and purity. In testing osmose juice, cool it down to normal temperature and determine the Brix by means of a spindle. Now dilute a small portion of the sample with pure water to about 17° Brix, take accurate Brix, determine the polariscope reading by the 110 c.c. method, and find the purity in Table IV. The Brix of the undiluted solution multiplied by the purity will give the sugar in the undiluted solution. In testing osmose waste water, cool down to normal temperature, take the Brix, polarize by the 110 c.c. volume method, and find the sugar and purity in Table V.

Calculation of Sugar Lost in Osmose Waste Water in Per Cent to the Weight of Beets.—The loss of sugar through the osmose process may be calculated from the loss of the dry substance of molasses through the process and purity of osmose waste water. The loss of the dry substance of molasses in the osmose waste water may be calculated by the formula,

$$y = \frac{m(P-a)}{P-P'}$$

which may be derived from the following data:

m—quantity of dry substance of molasses,

x—quantity of dry substance of osmosed molasses recovered from m quantity of dry substance of molasses,

y—quantity of dry substance of osmose waste water lost from m quantity of dry substance of molasses,

P-polarization of dry substance of osmosed molasses,

P'-polarization of dry substance of osmose waste water,

a-polarization of dry substance of original molasses.

Since the quantity of dry substance of the molasses is the sum of the dry substances of osmosed molasses and osmose waste water, we have

(1) 
$$m=x+y$$
; hence,  $x=m-y$ .

And since the polarization of the dry substance of molasses is the sum of polarizations of the dry substances of osmosed molasses and osmose waste water, we have

(2) 
$$ma=Px+P'y$$
.

Substituting m-y for x in the second equation in order to eliminate x, we have

$$ma = P(m-y) + P'y,$$

$$ma = Pm - Py + P'y,$$

$$Pm - ma = Py - P'y,$$

$$m(P-a) = y(P-P'),$$
therefore,  $y = \frac{m(P-a)}{P-P'}.$ 

Since the polarization of the dry substance is identical with the purity coefficient, the purities of molasses, osmosed molasses, and osmose waste water obtained from laboratory analysis may be used for a, P, and P' respectively. The quantity of dry substance of molasses m may be obtained by multiplying the quantity of original molasses by the Brix degree thereof. The calculation may best be shown by an example; assume that 305,684 kg. molasses of 80° Brix has been osmosed and that a=60.48, P=70.21, P'=38.85; then  $m=305,684 \times 0.8=244.547$  kg.,

and 
$$\frac{P-a}{P-P'} = \frac{70.21-60.48}{70.21-38.85} = 0.3103$$
.

The dry substance lost in osmose waste water y would then be  $244,547\times0.3103=75,883$  kg. Sugar lost in osmose waste water may be obtained by multiplying the dry substance lost in osmose waste water by the purity thereof, thus:  $y\times P'=75,883\times0.3885=29,480$  kg. To obtain sugar lost in osmose waste water in per cent to the weight of beets, sugar lost in osmose waste water in kg. should be divided by the weight of beets in kg. and multiplied by 100.

Principle of Steffen's Process.—As stated under CHEMICAL Properties of Cane-sugar, sugar forms compounds with lime which are insoluble in water. Steffen's process is an application of this principle. In this process, molasses is diluted with water to 12° Brix and cooled down to 15°C (50°F). The Brix of the solution should neither be much higher nor lower than 12°, as higher Brix makes the mixture of syrup and lime filter difficultly and lower Brix would unnecessarily increase the water consumption and require more time in filtering. When the desired temperature is reached, some powdered lime is added to the solution and mixed, whereupon the temperature rises owing to the action of the water on the lime. After each addition of lime, the temperature is brought down to about 15°C, as higher temperature tends to dissolve some of the saccharates of lime. As soon as the solution is cooled down to the desired point, some more lime is added and the temperature is lowered again. This operation is continued until a Brix spindle indicates 6.5 in the filtrate from the mixture of syrup and lime.

Apparatus for Steffen's Process.—The apparatus used for Steffen's process is called a Cooler. Fig. 63 represents an elevation view of the apparatus. The cooling chamber of this apparatus is similar to the heating chamber of the evaporator. J is the syrup space; A, valve for admitting water into the cooling chamber; A', outlet valve for cooling water; W, valve for admitting water into the syrup space; S, syrup inlet valve; M, agitator; P, propeller; C, outlet cock; L, lime hopper; T, lime measurer. The lime measurer is attached to the base of the lime hopper. It is simply a drum open at the upper side for receiving lime from the hopper and also at the base for discharging lime into the syrup chamber. Four wings attached to a shaft which passes through the center of the drum divide the interior space of the drum into 4 equal parts and each quadrant measures about 40 pounds of lime powder; hence, one-quarter turn of the shaft will discharge about 40 pounds of powdered lime into the juice chamber. The cooler is also provided with a thermometer and sample cock.

Operation of Cooler.—Open A and A' so that a current of cold water will be passing through the cooling chamber constantly, and start the agitator. Fill the syrup chamber with water so full as to cover the top of the cooling chamber, then

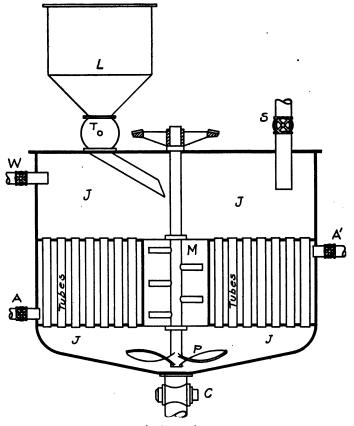


Fig. 63.—Cooler.

introduce a weighed quantity of molasses,—about 2500 pounds. After the solution is well mixed, draw a small portion of the solution through the sample cock and determine the Brix thereof by means of a Brix spindle. Should the solution be higher or lower than 12° Brix, it should be corrected by adding water or syrup as the case requires. This diluted molasses is called

Cooler Solution. As soon as the temperature of the solution is lowered to 15°C (59°F), commence adding lime by turning the shaft of the lime measurer one-quarter at a time. It is necessary to measure lime powder as the knowledge of the proportion of lime to the weight of molasses as well as to that of beets is very important in controlling Steffen's process. The weight of lime may be obtained by weighing the amount of lime measured by each quadrant section of the lime measurer. After a few minutes, when the temperature of the solution is lowered to a desired degree, add lime again. Continue this operation until from 35 to 40 quadrants of lime are added, keeping the temperature of the solution always below 21°C (70°F). Now draw a small portion of the limed molasses solution through the sample cock. filter, and determine the Brix degree of the filtrate by means of a Brix spindle. If it indicates about 6.5°, the operation should be terminated. If it is higher than 6.5°, continue adding lime. When the operation is complete, open the outlet cock C and pump the limed molasses solution up to filter presses, the principle and construction of which are identical with those of the first and second saturation juice filter presses. The deposit remaining in the frames of the filter presses is a compound of lime and sugar consisting of mostly di- and tri-calcium saccharates. The liquid flowing out through the cocks is waste water consisting of water, a small quantity of mono-calcium saccharate and impurities present in molasses. This liquid is known as Steffen's Waste Water and in most sugar-houses is run into the sewer. Continue filling one press until the pressure gauge indicates about 45 pounds, then fill another filter press and continue in this manner until all the limed molasses solution is pumped out from the cooler. A considerable quantity of impurity remains with the deposits adhering to them. In order to obtain purer saccharate, it is necessary to wash the deposits with cold water. As soon as the limed molasses solution is turned off, admit water into the the filter presses and continue washing until a Brix spindle indicates 2.0° in the wash water. The latter part of the wash water contains much less impurities and a quantity of sugar worth sav-

As soon as the Brix is brought down to 2.5°, the wash water is saved in a storage tank and used for diluting molasses in the cooler. When washing is completed, close the water-valve and open the compressed air valve so that the excess of water remaining with the deposits may be blown out by compressed air. The deposit of saccharate thus obtained is called "Saccharate Cakes." Steffen's filter presses are emptied in the same manner as the first and second saturation juice filter presses. Saccharate cakes are run into a mixing tank, mixed with the sweet water from the first and second saturation juice filter presses until a Baumé spindle indicates 16 degrees in the mixture and are used for defecating diffusion juice. The mixture is called "Milk of Saccharate." When there is no diffusion juice to be defecated, milk of saccharate alone may be saturated, reducing the alkalinity to almost neutral. The juice obtained from saccharate may be concentrated the same as thin juice.

Chemical Examination of Products from Steffen's Process.— It is very important to chemically examine the initial molasses, lime, and various products from Steffen's process in order to successfully control the operation of this process. The only means for detecting whether or not the process is properly operated is the chemical examination of the products, and the success or failure of the process largely depends upon the accuracy of the chemical work.

Initial Molasses.—A small portion of molasses should be saved every time the molasses is introduced into the cooler in order to obtain an average sample. About 4 analyses should be made every 24 hours. Brix, sugar, and purity should be determined in the same manner as those of fillmass (p. 239).

Powdered Lime.—Lime should be examined for grit and calcium oxide. A sample should be taken every 30 minutes and saved in order to obtain an average sample. About 4 analyses in 24 hours would be sufficient.

Grit Test.—Weigh out exactly 50 grams of lime powder, sift through a 60-mesh sieve until no more particles pass through,

weigh the residue remaining in the sieve and multiply the weight by 2. The product is the per cent of grit.

Calcium Oxide.—Mix the sample thoroughly, take a small portion, grind it in a mortar as fine as possible, weigh out exactly 0.5 gram, transfer to a porcelain evaporator or glass beaker, mix with a little water, add a few drops of phenolphthalein solution, and titrate with the standard sulphuric acid solution, I c.c. of which neutralizes 0.01 gram of calcium oxide. The titration should be continued until the pink color of the solution no longer returns. The number of c.c. of the solution required multiplied by 2 will give the per cent of calcium oxide.

Cooler Solution.—Cooler solution should be examined for Brix, sugar, and purity. A small portion of the cooler solution should be saved from every cooler in order to obtain an average sample. Mix the sample thoroughly, pour into a glass cylinder, determine the Brix, correct for temperature if necessary, fill a 110 c.c. flask to the 100 c.c. mark with the solution, add a few drops of phenolphthalein solution, mix by shaking, neutralize with strong acetic acid by adding the acid a little at a time, shaking continually until the pink color of the solution just disappears, then complete the volume to 110 c.c. with dilute lead subacetate solution, mix thoroughly, filter, polarize, and find the sugar and purity in Table IV.

Saccharate.—Saccharate is generally examined for sugar, purity, and alkalinity. A sample of saccharate should be taken from the saccharate filter presses in the same manner as lime cakes.

Sugar.—Mix the sample thoroughly, weigh out one-half normal weight, transfer completely to a 100 c.c. flask with as little water as possible, add a few drops of phenolphthalein solution, neutralize with strong acetic acid by adding the acid a little at a time, shaking continually until the pink color of the solution just disappears, cool the flask and content in cold water, add 5 c.c. of dilute lead solution, complete the volume to 100 c.c. with

water, mix thoroughly, filter, and polarize. The polariscope reading multiplied by 2 gives the sugar content in per cent.

Purity.—Mix about 2 kg. of the sample with 2 liters of pure water in a small tank, pass carbon dioxide gas until a proof no longer gives a pink color with phenolphthalein solution, filter, evaporate the filtrate to about 15° Brix on a hot water bath, or by a suitable means, filter again, cool, determine the Brix, fill a 110 c.c. flask to the 100 c.c. mark, add a few drops of lead solution, make up to the 110 c.c. mark with water, mix thoroughly, filter, polarize, and find the purity in Table IV.

Alkalinity.—The alkalinity of saccharate is determined in order to estimate the available calcium oxide therein. Weigh out exactly I gram, transfer to a porcelain evaporator, add a few drops of phenolphthalein solution, titrate with the standard sulphuric acid solution I c.c. of which neutralizes 0.01 gram of calcium oxide until the pink color just disappears. Allow it to stand for one-half hour, stirring occasionally; should a pink color return, as is usually the case, carefully titrate it again until the pink color just disappears. The burette reading directly gives the alkalinity or available lime in per cent.

Steffen's Waste Water and Wash Water—Steffen's waste water and wash water should be examined for Brix and sugar. Waste water should be saved from every cooler made and the wash water be taken from the wash water storage tank in order to obtain an average sample. Polarization and Brixing of these products are carried out exactly in the same manner as those of the cooler solution.

Having obtained the polariscope reading for waste water, the sugar content therein may be found in the following table:

	.o	.ı	.2	.3	.4	-5	.6	•7	.8	.9
o	.00	.03	.06	.08	.11	.14	17	.20	.22	.25
I	.28	.31	.34	.36	•39	.42	.45	.48	.50	∙53
2	.56	.59	.62	.64	.67	.70		.76	.78	.8r
3	.84	.87	.90	.92	-95	.98	1.01	1.04	1.06	1.09
4	1.12	1.15	1.18	1.20	1.23	1.26	1.29	1.32	1.34	1.37
5	1.40	1.43	1.46	1.48	1.51	1.54	1.57	1.60	1 .62	1.65

From the polariscope reading for wash water, the sugar content therein may be found in the following table:

	.0	.I	,2	-3	.4	-5	.6	.7	.8	.9
0	.00	.03	.06	.08	.11	.14	.17	.20	.22	.25
I	.28	.31	.34	.36	.39	.42	.45	.48	.50	• •53
2	-57	.60	.63	.65	.68	.71	.74	.77	.79	.82
3	.85	<b>.8</b> 8	.91	-93	.96	.99	1.02	1.05	1.07	1.10
4	1.13	1.16	1.19	1.21	1.24	1.27	1.30	1.33	1.35	1.38

The direction for using these tables is given under ESTIMATION OF SUGAR IN PULP.

Calculation of Sugar Lost in Steffen's Waste Water in Per Cent to the Weight of Beets.—It has been found by long experience that 34 per cent of sugar in the dry substance of molasses is carried away by the waste water; hence, when

d=dry substance of molasses worked in kg.

P=purity of molasses

b=weight of beets worked in kg.

Sugar lost in Steffen's waste water in per cent to weight of

beets = 
$$\frac{d \times P \times 0.34 \times 100}{b}$$
.

Hot Process.—Steffen's waste water contains some sugar and lime, probably in the form of mono-calcium saccharate. This soluble saccharate of lime may be precipitated by heating to boiling, the resulting product being tri-calcium saccharate. The process of recovering sugar from Steffen's waste water by heating is called Steffen's Hot Process. The apparatus used for heating Steffen's waste water is an ordinary iron tank provided with steam-coils. Hot saccharate is filtered and washed in the same manner as cold saccharate. Saccharate, waste water, and wash water from the hot process are chemically examined in the same manner as those from the cold process.

## Chapter IX.

## SPECIAL ANALYSES.

There are various special analyses that the sugar-house chemist is called upon to make from time to time. In this work, methods for the more important analyses will be described.

Determination of Water and Total Solids in Juice, Fillmasses, and Syrups.—As stated previously, the Brix spindle does not indicate the true total solids in the juices or syrups, owing to the fact that these products contain substances other than canesugar, whereas the Brix spindle is graduated according to pure sugar solutions. In order to obtain true total solids in these products, it is necessary to determine the amount of water present by drying a sample.

Place 6 or 7 grams of pure quartz sand and a short stirring rod, glass or platinum, in a flat-bottomed porcelain or platinum dish, ignite over a direct flame or in a muffle, cool in a desiccator, and weigh accurately. Now mix the sample to be examined thoroughly and place 3 or 4 grams thereof in the dish, mix carefully with the sand and weigh the whole accurately; dry in a hot water oven for from 10 to 12 hours, stirring at intervals of one hour; cool in a desiccator, and weigh. After noting the weight, stir, dry again in a hot water oven for one hour, cool, and weigh again. Repeat the drying and weighing until the difference between the two weighings does not exceed 2 mg.

The loss in weight  $\times$  100
The weight of sample taken = per cent water; 100—per cent water = per cent true total solids.

True purity is obtained by dividing the per cent of sugar found in polarization by the total solids and multiplying by 100:

 $\frac{\text{Per cent sugar} \times 100}{\text{Per cent total solids}} = \text{true purity.}$ 

Determination of Ash.—Ignite the residue from the determina-

tion of total solids in a muffle, cool in a desiccator, and weigh. Repeat until a constant weight is obtained. The final weight minus the weight of the sand and dish will give the weight of the ash.

The weight of  $ash \times 100$ The weight of sample taken = per cent ash.

Saline Coefficient.—The saline coefficient is the quantity of sugar per unit of ash and may be calculated as follows:

Per cent sugar Per cent ash = saline coefficient.

Coefficient of Organic Matter.—The coefficient of organic matter is the quantity of sugar per unit of organic matter and may be calculated as follows:

Per cent sugar
Per cent total solids—per cent sugar—per cent ash
of organic matter.

Saline and organic coefficients are interesting to know owing to the fact that a definite quantity of these non-sugars prevents a definite quantity of sugar from crystallization.

Determination of Ash as Sulphate.—There is another method used for determination of the ash in fillmasses and syrups by the aid of sulphuric acid. In this method, however, the weight of ash is increased by I/I0 its own weight, owing to the conversion of carbonates into sulphates; therefore, the weight of ash obtained as sulphate ash must be reduced to carbonate ash by multiplying by 0.9. Ignite a flat-bottomed porcelain or platinum dish, cool in a desiccator, and weigh. Place about 3 grams of the sample to be examined in the dish and weigh accurately. Add about 15 drops of pure concentrated sulphuric acid and heat cautiously over a direct flame until the acid is driven off and intumescence ceases, then ignite in a muffle at low redness until the residue becomes white or nearly so. When ignition is complete, remove the dish from the muffle, cool in a desiccator, and weigh;

The weight of sulphate  $ash \times 0.9 \times 100$ The weight of sample taken

Determination of Total Lime.—As stated under Sugar-Boil-Ing, lime is present in the beet-juice sometimes in the form of neutral salts of organic acids and causes difficult boiling. Lime in such a form could not be estimated by the alkalinity method, as it has no alkalinity reaction. In such a case, it is necessary to determine the total lime. The total lime minus the alkaline lime would be equal to the lime existing in the form of salts. The accurate determination of total lime is best made by the gravimetric method, which will be described under Limestone Analysis. The gravimetric method, however, requires long manipulation and is not practical in daily sugar-house work where rapid determination is required. A method which is very quick and sufficiently accurate for daily sugar-house work is that of Soap Solution.

Preparation of Soap Solution.—Dissolve 20 grams of pure caustic potash in 25 c.c. of pure water, mix with 200 c.c. of strong alcohol, add 100 c.c. of pure olive oil, warm the mixture on a water bath in a flask provided with a return condenser until saponification is complete. The completeness of saponification may be found by introducing a drop of the solution into pure water, where it will dissolve completely, leaving no turbidity when the oil is completely saponified. Mix the solution with about 3 liters of pure water, add a solution of calcium chloride, a little at a time, stirring constantly as long as a precipitate forms, filter, wash the precipitate thoroughly. After expelling water adhering to the precipitate, as much as possible, by gentle pressure, mix the precipitate with about 40 parts (grams) of potassium carbonate, and extract repeatedly with 96 per cent alcohol, in a flask provided with a return condenser, on a hot water bath. The extracts are mixed and filtered. The soap solution thus obtained should be diluted and standardized to such a strength that I c.c. of the solution will correspond to 0.001 gram of calcium oxide.

Standard Hard Water.—In order to standardize soap solu-

tion, it is necessary to prepare a solution of calcium chloride of such strength that I c.c. will correspond to 0.0001 gram of calcium oxide. Weigh out exactly 0.1786 gram of pure calcium carbonate (Iceland spar), dissolve in a few c.c. of dilute hydrochloric acid, evaporate to dryness, dissolve in water, and make up exactly to 1000 c.c. with water at the temperature of the room. I c.c. of this solution corresponds to 0.0001 gram of calcium oxide. An equivalent solution of barium chloride may be prepared by dissolving 0.436 gram of pure barium chloride in exactly I liter of pure water.

Standardization of Soap Solution.—Fill a 50 c.c. burette with the soap solution exactly to the zero mark; transfer by means of a pipette 40 c.c. of standard hard water to a glass-stoppered bottle of 200 c.c. capacity with a 100 c.c. graduation mark and fill up to the 100 c.c. mark with water. Now add the soap solution from the burette, a little at a time, and shake vigorously after each adding. Toward the end of titration, add one drop at a time. Continue this operation as long as the foam formed on the surface of the liquid disappears quickly. When the foam remains permanent or for 5 minutes, the titration is complete. Note the burette reading. Should the soap solution be of standard strength, that is, of such strength that I c.c. is equivalent to 0.001 gram of lime, 4 c.c. would be required to titrate 40 c.c. standard hard water. Should the burette reading in the titration just made be 1.6 c.c., it is evident that 1.6 c.c. of the soap solution must be diluted to 4 c.c. Larger quantity of the soap solution must be diluted in the same proportion; for example, 400 c.c. of the soap solution must be diluted to 1000 c.c. Repeat the operation and take the average result for final value. Dilution should be made with 60 per cent alcohol.

Determination of Total Lime in Juices by Means of Soap Solution.—Transfer by means of a pipette, 10 c.c. of the juice to be examined to a titration bottle, fill with water to the 100 c.c. mark, and titrate with the standardized soap solution according to the direction described above. Assuming that 10 c.c. of the juice required 2.9 c.c. of the soap solution, the total lime

contained in 10 c.c. of the juice would be  $0.001\times2.9=0.0029$  gram. The per cent of total lime would then be  $\frac{0.0029\times100}{10}=0.029$ .

Determination of Total Alkalinity and Alkalinity Due To Caustic Soda and Potash.—Alkalinity of juice or syrups is caused not only by calcium oxide but also by hydroxides of sodium and potassium which were liberated by the action of lime on the organic salts of those bases. It is sometimes desirable to determine both the total alkalinity and the alkalinity due to caustic soda and potash.

Total Alkalinity.—The total alkalinity of a light-colored substance may be determined by the usual method. When the substance to be examined is very dark, a small quantity must be taken; weigh out exactly 2 grams of the substance to be examined, dissolve in cold, recently boiled, distilled water, transfer to a 100 c.c. flask, make up to the mark, and mix thoroughly. Transfer exactly one-half of this solution to a small porcelain evaporator and the other half to another evaporator of the same size, add a few drops of phenolphthalein solution to one dish and keep the other dish for comparing the color of the solutions without adding any indicator. Titrate the solution which was mixed

with phenolphthalein with the  $\frac{N}{28}$  sulphuric acid solution until the color of the solution becomes nearly the same as that of the solution in the other dish. Under these conditions, the number of c.c. of the acid required divided by 1000 and multiplied by 1000 would be the alkalinity in per cent, expressed in terms of lime.

Alkalinity Due to Caustic Alkalies.—Treat a certain volume of juice to be examined with an equal volume of strong alcohol, whereupon caustic lime will be precipitated as insoluble saccharate, filter, and titrate 10 c.c. of the filtrate the same as usual. The number of c.c. of the acid taken multiplied by 2 and divided by 100 would be the alkalinity due to caustic alkalies in per cent, expressed in terms of lime.

Determination of alkalinity due to caustic alkalies in dark sub-

stances may be conducted in the same manner after diluting a weighed quantity of the substance to a definite volume with pure water

Buisson's Method for Determining Alkalinity in Highly Colored Substances.—Transfer 25 c.c. of the solution of the substance to be examined to a glass-stoppered bottle, add one drop of neutral solution of corallin, and 10 c.c. of neutral ether. Now add the standard acid solution a little at a time. After each adding, shake vigorously and allow to stand for a few seconds so that the ether may separate and rise to the surface of the solution. The slightest excess of acid turns the color of the ether yellow.

Determination of the Volume of Lead Precipitate.—Add subacetate of lead to 100 c.c. of the solution to be examined, placed in a tall cylinder, until no more precipitate falls, wash the precipitate by decantation, using hot water, until all sucrose is removed. Transfer the precipitate to a 100 c.c. flask, add one-half normal weight of pure cane-sugar, make up to the graduation mark with water, mix thoroughly, filter, and polarize in a 400 m.m. tube. Assuming that the sugar polarizes 99.9 without lead precipitate and 100.77 in the presence of lead precipitate, the volume occupied by lead precipitate would be

$$100 - (100 \times \frac{99.9}{100.77}) = \frac{(100 \times 100.77) - (100 \times 99.9)}{100.77} = 0.86 \text{ c.c.}$$

Determination of Reducing Sugars: Invert Sugar.—The term reducing sugar is applied to a class of sugars which have the power to reduce copper or certain other metals in an alkaline solution. As a definite quantity of any of these sugars reduces a definite quantity of copper, their quantity may be determined by estimating the quantity of copper they reduce. Invert sugar belongs to this class and may be determined by means of alkaline copper solution.

Invert sugar disually occurs in sugar-beets in more or less quantities according to the quality of beets. Invert sugar in beets is usually decomposed during defectaion with lime or concentration. The presence of a large quantity of invert sugar

in the after-products usually indicates defective operations, for inversion takes place when the juice is saturated with sulphur gas to acidity or when the juice becomes acid on evaporation. It is evident, then, that the estimation of invert sugar in after-products gives important bearings in controlling the sugar-house operation.

Reagents for Determination of Invert Sugar.—The reagent required for determining invert sugar is Fehling's Solution. This reagent is made in two solutions, since it suffers decomposition when made in one solution. One solution is designated by F. I, the other by F. II. F. I is made by dissolving 34.63 grams of pure copper sulphate in 500 c.c. of pure water; F. II is made by dissolving 173 grams of pure Rochelle salt and 50 grams of pure caustic soda in 500 c.c. of pure water.

The Method for Determination of Invert Sugar in a Material Containing I Per Cent or Less of Invert Sugar and a High Percentage of Sucrose.—Weigh out exactly 20 grams of the material to be examined in a sugar-dish, dissolve with water, transfer to a 100 c.c. flask, clarify with lead subacetate solution, complete the volume to the graduation mark with water, mix thoroughly, and filter. Remove the excess of lead in the filtrate by adding dry sodium carbonate until no more precipitate falls and by filtering the mixture. Place 50 c.c. of the filtrate containing 10 grams of the substance taken in a beaker, add 25 c.c. F. I and 25 c.c. F. II. Now heat cautiously over a direct flame, taking about 4 minutes to reach the boiling point, and boil for exactly 2 minutes. After the completion of boiling, add 100 c.c. of cold, recently boiled, distilled water and allow it to stand for a few minutes in order that the reduced cuprous oxide may settle down, then filter, and wash until the last filtrate contains no trace of copper which may be tested by potassium ferrocyanide. When the washing is complete, place the filter-paper and cuprous oxide in a weighed small porcelain crucible, dry, incinerate the filter-paper, ignite the whole at low redness, cool, and weigh. Repeat the igniting and weighing until all the cuprous oxide is converted into cupric oxide and consequently a constant weight is obtained. The weight of cupric oxide multiplied by 0.799 will give the weight of metallic copper. When rapid determination is desired, dissolve the precipitate of cuprous oxide in nitric acid and determine the copper by a standard potassium cyanide solution according to the direction given under COPPER. Then find, in Herzfeld's table, the per cent of invert sugar corresponding to the weight of metallic copper.

Copper reduced by 10 grams substance mg.	Invert sugar per cent	Copper reduced by 10 grams substance mg.	Invert sugar per cent	Copper reduced by to grams substance mg.	Invert sugar per cent
50	.05	120	.40	190	.79
55	.07	125	•43	195	.82
60	,09	130	.45	200	.85
6 <b>5</b>	.II	135	.48	205	.88
70	. 14	140	.51	210	.90
<b>75</b> °	. 16	145	∙53	215	.93
8o	.19	150	.56	220	.96
85	.21	155	.59	225	.99
90 ·	.24	160	.62	230	1.02
95	.27	165	.65	235	1.05
100	.30	170	.68	240	1.07
105	.32	175	.71	245	1.10
110	·35	180	•74		
115	.38	185	.76		

Factors for Conversion of Metallic Copper Into Invert Sugar in Material Containing More Than I Per Cent of Invert Sugar.

—The factors for conversion of metallic copper into invert sugar are variable, according to the proportion of invert sugar; for example, when the proportion of sucrose to invert sugar is 10 to 90 and the absolute weight of invert sugar is approximately 200 mg., the factor is 56.3. On the other hand, when the proportion of sucrose to invert sugar is 99 to I and the approximate absolute weight of invert sugar is 50 mg., the factor is 38.1. For this reason, it is absolutely necessary, in order to obtain correct results, that some empirically established factors be used for the interpretation of analytical data. The following table has

been prepared by Meissl and Hiller, giving the factors for the various proportions of sucrose to invert sugar and the various absolute weights of invert sugar. R represents sucrose; I, invert sugar; Z, approximate absolute weight of invert sugar in milligrams; F, factors in per cent.

Ratio of		_		milligram	ıs.		
R to I	200	175	150 F i	n per cent	. 100	75	50
0:100	56.4	55-4	54.5	<b>5</b> 3.8	53.2	5 <b>3.0</b>	53.0
10:90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20:80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30:70	56. I	55.1	54.2	53-7	53.2	52.6	52.6
40:60	55.9	55.0	54. I	53.6	53.1	52.5	52.4
50:50	55.7	54.9	54.0	53.5	53.0	52.3	52.2
60:40	55.6	54-7	53.8	53.2	52.8	<b>52.</b> I	51.9
70:30	5 <b>5</b> ·5	54.5	53.5	52.9	52.5	51.9	51.6
80:20	55-4	54.3	53.3	52.7	52.2	51.7	51.3
90:10	54.6	53.6	53.1	52.6	<b>52.</b> I	51.6	51.2
91:9	54.I	53.6	52.6	52.1	51.6	51.2	50.7
92:8	53.6	53. I	<b>52.</b> I	51.6	51.2	50.7	50.3
93:7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94:6	53.I	52.6	51.6	50.7	50.3	49.8	48.9
95:5	52.6	<b>52.</b> I	51.2	50.3	49.4	48.9	48.5
96:4	<b>52.</b> I	51.2	50.7	49.8	48.9	47.7	46.9
97:3	50.7	50.3	49.8	48.9	47.7	46.2	45.I
98:2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99:1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

Method for Determination of Invert Sugar in Material Containing More Than I Per Cent Invert Sugar.—Weigh out 2 normal weight of the sample in a sugar-dish, mix with water, transfer to a 200 c.c. flask, clarify with lead, make up to the graduation mark with water, mix, filter, polarize, and note the polariscope reading. Remove the excess of lead in the remainder of the filtrate by adding dry sodium carbonate or sulphate in a slight excess and by filtering. Prepare a series of solutions in large test-tubes by placing I, 2, 3, 4, 5 c.c. of this filtrate successively, add 5 c.c. of Fehling's solution prepared by mixing equal volume of F. I and F. II to each test-tube and heat to boiling; continue to boil for two minutes, and filter. Observe which solution has the lightest but still a distinctly blue tint and note the number

of c.c. of that solution. Transfer 20 times this volume of the sugar solution to a 100 c.c. flask, make up to the graduation mark with water and mix thoroughly; use 50 c.c. of this solution for determination of invert sugar, using 25 c.c. F. I and 25 c.c. F. II, boiling, and estimating the copper in the same manner as in the method for determination in the material containing less than I per cent invert sugar. Assuming that

Cu=the weight of copper obtained,

P=the polariscope reading,

W=the weight of the sample in 50 c.c. of the solution used for the determination,

F=the factor obtained from Meissl and Hiller's table,

$$\frac{Cu}{2} = \text{the approximate absolute weight of invert sugar}$$

$$= Z,$$

Y=the approximate per cent of invert sugar,

R= the number for sucrose in the proportion of sucrose to invert sugar, and

I= the number for invert sugar in the proportion of sucrose to invert sugar,

it is apparent that

$$Y = \frac{100Z}{W}; R = \frac{100P}{P+Y}; 100 - R = I;$$

$$\frac{CuF}{W} = \text{per cent of invert sugar.}$$

$$Example-assume that P = 86.4,$$

$$W = 4.0 \text{ grams, } Cu = 0.304 \text{ gram, then,}$$

$$Z = \frac{Cu}{2} = \frac{0.304}{2} = .152 \text{ gram;}$$

$$Y = \frac{Z \times 100}{W} = \frac{0.152 \times 100}{4} = 3.8;$$

$$R = \frac{P \times 100}{P+Y} = \frac{86.4 \times 100}{86.4 + 3.8} = 95.8;$$

$$I = 100 - R = 100 - 95.8 = 4.2;$$

$$Hence, R : I = 95.8 : 4.2.$$

Consulting Meissl and Hiller's table, we will find the number for

the factor in the column under 150, the value nearest to 152, and opposite 96: 4, the value nearest to 95.8: 4.2. The number sought is 50.7. Hence, the per cent of invert sugar would be

$$\frac{\text{CuF}}{\text{W}} = \frac{0.304 \times 50.7}{4} = 3.85.$$

Determination of Sucrose in the Presence of Reducing Sugars.—The presence of an appreciable quantity of invert sugar in a solution to be examined for sucrose would interfere with the polarization of the latter, owing to the fact that the former rotates the plane of polarized light to the left. It is necessary then, in determining sucrose by the optical method, in the presence of an appreciable quantity of invert sugar, to employ a method that eliminates this error. Such a method has been devised by Clerget. It is called the *Inversion Method*.

Pure sucrose when polarized in a 200 m.m. tube at 0°C, rotates 100° to the right before inversion and 42.66° to the left after inversion. The total rotation at 0°C would then be 142.66°. Actual observation, however, is usually made at 20°C or thereabouts, as it is not practicable at 0°C. The rotatory power of invert sugar diminishes with the rise of temperature by 0.5 degree per 1°C, hence, the total rotation at a temperature other

than o°C would be 142.66 
$$-\frac{t}{2}$$
; for example, 142.66  $-\frac{20}{2}$ 

= 132.66 when polarized at 20° C.

Polarization before inversion is called *Direct Polarization* and designated by +P. Polarization after inversion is called *Invert Polarization* and designated by -I. The algebraic sum of the two polarizations would be +P-(-I)=P+I. This is called *Clerget's Sum* and designated by S. This sum could not be influenced by the presence of invert sugar as the optical activity of invert sugar remains the same before and after inversion. Taking the long confirmed figure 132.66 for the total rotation of pure sucrose at 20°C and S for the total rotation of the substance examined at the same temperature, the per cent of sucrose in the latter may be calculated from the following proportion:

132.66 : 100 :: S : per cent sucrose ; hence, per cent sucrose =  $\frac{100S}{132.66} = 0.7538S.$ 

Method for Invert Polarization.—Dissolve one-half normal weight, or 13.024 grams, of the substance to be examined with 75 c.c. of water in a 100 c.c. flask and add 5 c.c. of hydrochloric acid solution of specific gravity 1.188, or containing 38 per cent of the acid. After mixing the content by a circular motion, place the flask in a water bath heated to 70°C. temperature of the solution should be so controlled that it would reach 67° to 70°C in two or three minutes. Maintain the temperature of the solution at 60°C as nearly as possible 5 minutes longer, mixing the contents of the flask constantly by a circular motion. The duration of heating should not exceed ten minutes under any circumstances. At the end of the time specified, remove the flask from the water, cool down to 20°C, make it up to 100 c.c. with water, mix, and filter. Should the color of the solution be too dark for polariscopic observation, add I gram of bone-black before filtering. Fill a 200 m.m. observation tube, provided with a water-jacket and a thermometer, with the solution, place the tube in the receptacle of a double compensation polariscope, pass a current of water at about 10°C into the waterjacket by means of rubber tubing arranged in a syphon. As soon as the temperature of the solution reaches 20°C as indicated by the thermometer, take accurate reading. Read several times and take the average.

Direct polarization may be carried out in the usual manner, excepting that it should also be read at 20°C in a tube provided with a water-jacket and a thermometer.

## EXAMPLE.

Direct polarization	• • • •	 • • • •	 	• • • • • •	14.8
Invert polarization		 • • • •	 	—	12.7
Temperature		 	 		20°C

As -12.7 is the polarization for one-half normal weight, polarization for one normal weight would be  $-12.7 \times 2 = -25.4$ , and the Clerget's sum would be 14.8 + 25.4 = 40.2; hence,

per cent sucrose=40.2×0.7538=30.3.

Determination of Raffinose and Sucrose.—The disturbance caused by the presence of raffinose in saccharine substances is two-fold; one is the modification of the crystal forms of sugar, the other is its optical activity causing too high results in the analysis of saccharine substances, since its rotatory power is 1.85 times as great as that of sucrose. It is necessary, therefore, to examine fillmass, syrups, and molasses from time to time for raffinose.

There are several formulas used for calculating sucrose and raffinose from direct and invert polarizations. The one most commonly adopted is that of Herzfeld. In this formula, direct polarization is designated by P; per cent of sucrose, by S; per cent of raffinose, by R; invert polarization, by I. It has been observed that, in direct polarization, the rotation of anhydrous raffinose is 1.85 times that of pure sucrose and also that, after inversion, 26.048 grams of pure sucrose rotate at 20°C 32.66° to the left and the same weight of anhydrous raffinose rotates, at the same temperature, 94.9° to the right; then

(1) 
$$P=S+1.85R$$
;

(2) 
$$I = -0.3266S + 0.949R$$
;

from the second equation we derive

$$R = \frac{I + o.3266 \, S}{o.949};$$

substituting this value in the first equation in order to eliminate R, we derive

$$P = S + 1.85 \left( \frac{I + 0.3266S}{0.949} \right);$$

$$S = \frac{0.5124P - I}{0.839}.$$

hence,

From the first equation, we derive directly

$$R = \frac{P - S}{1.85}.$$

Inversion and polarization in the Herzfeld method are conducted in exactly the same manner as in the Clerget method.

### EXAMPLE.

$$S = \frac{0.5124 \text{ P} - \text{I}}{0.839} = \frac{0.5124 \times 96 - (-30)}{0.839} = 94.39;$$

$$P - S = 96.0 - 94.39$$

$$R = \frac{P - S}{1.85} = \frac{96.0 - 94.39}{1.85} = 0.87.$$

Determination of Raffinose and Sucrose in the Presence of Invert Sugar.—Herzfeld's formulas for sucrose and raffinose could not be used when an appreciable quantity of invert sugar is present in the substance to be examined, as invert sugar is levo-rotatory. For such cases, Wortmann has elaborated a method based on the following considerations. In Wortmann's formula, P and P' represent respectively direct and invert polarizations of normal weight of the substance examined; N, the per cent of invert sugar determined by means of Fehling's solu-

tion and calculated by the formula  $\frac{Cu\times47}{Q}$ , in which Q repre-

sents the amount of the substance taken, Cu, the amount of copper reduced, and 47, the average factor of the Meissl table; Z represents the per cent of sucrose, R, the per cent of raffinose. Since 26.048 grams of invert sugar rotate 31.03° to the left on the Ventzke scale when polarized in a 200 m.m. tube at 20°C, the rotation factor of invert sugar would be —0.3103. The invert rotation factors of sucrose and raffinose are respectively —0.3266 and +0.9598, as sucrose rotates —32.66° and raffinose, +95.98° after inversion. It is evident, then, that

(1) 
$$P = Z + 1.85R - 0.3103N$$
;

(2) 
$$P' = -0.3266Z + 0.9598R - 0.3103N$$
.

From the second equation, we derive

$$R = \frac{P' + 0.3266Z + 0.3103N}{0.9598};$$

substituting this value in the first equation in order to eliminate R, we obtain

$$P = Z + \frac{1.85 \times (P' + 0.3266Z + 0.3103N)}{0.9598} - 0.3103N,$$

from which we derive  $Z = \frac{0.9598P - 1.85P' - 0.277N}{1.5648}$ ;

from the first equation, we derive  $R = \frac{P - Z + 0.3103N}{1.85}$ .

### EXAMPLE.

 Direct polarization
 53.9

 Invert polarization
 7.4×2=−14.8

 Copper reduced by 10 grams
 0.346 gram

$$N = \frac{Cu \times 47}{Q} = \frac{0.346 \times 47}{10} = 1.63 \text{ per cent};$$

then, 
$$Z = \frac{0.9598 \times 53.9 - (1.85 \times -14.8) - 0.277 \times 1.63}{1.5648} = 50.27;$$

$$R = \frac{53.9 - 50.27 + 0.3103 \times 1.63}{1.85} = 2.24.$$

Per Cent of Crystallized Sugar.—It is often desirable to find the per cent of crystallized sugar in fillmass. Dupont devised a method which requires the following data:

P-the polarization of crystallized sugar,

P'—the polarization of syrups,

a—the polarization of fillmass,

m—the weight of fillmass,

x—the weight of crystallized sugar from m quantity of fillmass.

y—the weight of syrup from m quantity of fillmass.

Since it is evident that the polarization of fillmass is the sum of those of crystallized sugar and syrup and that the weight of fillmass is the sum of those of crystallized sugar and syrup, we have

(1) 
$$Px+P'y=ma;$$

$$(2) \quad x+y=m;$$

hence.

$$y = m - x$$
;

substituting m-x for y in the first equation in order to eliminate y, we derive

$$Px+P'(m-x) = ma,$$

$$Px+P'm-P'x = ma,$$

$$Px-P'x = ma-P'm,$$

$$x(P-P') = m(a-P'),$$

$$x = \frac{m(a-P')}{P-P'};$$

therefore.

$$x=\frac{m(a-r)}{P-P'};$$

when m=1 and crystallized sugar is pure or P=100, we have

$$x = \frac{a - P'}{100 - P'}$$
 and  $y = 1 - x$ .

Method for Determination of Crystallized Sugar.—Heat about 500 grams of fillmass to be examined to 85°C, separate the syrup by means of a small centrifugal machine constructed for laboratory purposes, dry the sugar crystals as thoroughly as possible and determine the percentages of sucrose in the crystals and in the syrup by polarization. Let the data obtained be as follows:

then, 
$$a = 84.5$$
;  $P = 100$ ;  $P' = 60.6$ ;  $x = \frac{a - P'}{100 - P'} = \frac{84.5 - 60.6}{100 - 60.6} = 0.6066$ ;

hence, the percentage of crystallized sugar in the fillmass examined would be 100x = 60.66.

Determination of Marc.—Marc is the insoluble portion of beets. It is desirable to estimate the percentage of marc in beets occasionally in order to confirm the proportion of the juice to the insoluble matter in beets, or, the juice factor. The method described below is the simplest and is fairly accurate.

filter-paper in a drying bottle, dry one-half hour in a hot water oven, cool, and weigh accurately. Then weigh out accurately about 10 grams of finely ground beets, place in a beaker, wash with water at from 65° to 70°C, first by decantation, filter the wash water through the filter-paper just dried and weighed, in order to collect the fine particles of beets suspended in the water. Transfer the pulp on the filter-paper when nearly all the soluble matter is washed out and continue washing until the last filtrate no longer contains soluble matter, which

may be tested by evaporating a few drops in a clean watchglass. When the washing is complete, place the pulp and filterpaper in the drying bottle, dry in a hot water oven to constant weight. Designate the weight of the beets taken by P, the weight of the dry filter-paper by P', and the weight of the dry filterpaper and dry pulp by P"; then

per cent of marc = 
$$\frac{100(P''-P')}{P}$$
;

per cent of juice =  $100 - \frac{100(P''-P')}{P}$ .

Analysis of Limestone.—As stated under CALCIUM, the quality of limestone is a very important factor in sugar manufacture. The usual ingredients estimated in limestone are moisture, acid insoluble matter (mostly silica), iron oxide, alumina, calcium oxide, carbon dioxide, magnesia, and sulphuric anhydride.

Preparation of Sample.—Chip off small pieces from a large number of limestone rocks to be examined, reduce all the pieces to the size of a pea, mix the whole thoroughly, and obtain a small sample by quartering and requartering. Grind the sample thus obtained as fine as possible, sift the entire sample through an 80-mesh sieve, regrinding the residue in the sieve, should there be any, and mix thoroughly.

Determination of Moisture.—Weigh exactly 10 grams of the sample in a weighing bottle, dry in an air oven, heated to 120°C, to constant weight.

The loss in weight 
$$\times$$
 100 = per cent of moisture.

Determination of Acid Insoluble Matter.—Weigh out exactly I gram of the sample, dissolve in a few c.c. of dilute hydrochloric acid in a platinum dish, adding a very small quantity of the acid at a time, in order to prevent the loss by projection, evaporate to perfect dryness on a water bath, moisten the residue with a few drops of concentrated hydrochloric acid, evaporate to perfect dryness on a water bath again, heat the dish and contents in an air oven heated to 110°C for one hour, treat with dilute hydrochloric acid, filter through a small ashless filter-paper, wash

the residue in the filter-paper with hot water until the last filtrate no longer contains soluble matter, dry the filter-paper and contents, ignite in a weighed platinum crucible, cool, and weigh. The weight of the residue × roo= per cent of acid insoluble matter.

Determination of Iron Oxide and Alumina.—Collect all the filtrate and wash water from the determination of the acid insoluble matter in one beaker, boil, add ammonia in an excess, whereupon hydroxides of iron and aluminum will be precipitated. rapidly while hot and wash the precipitate with hot water thor-In case a considerable quantity of iron oxide and alumina is present, dissolve the precipitate by pouring a few c.c. of dilute hydrochloric acid over the filter-paper and wash thoroughly, reprecipitate the iron and the alumina in the filtrate with ammonia, collect the precipitate on the same filter-paper and wash thoroughly again. Dry the precipitate and filterpaper, incinerate the paper and ignite the whole in a weighed platinum crucible, cool, and weigh. The weight of the residue ×100= per cent iron oxide and alumina.

When present in abnormal quantity, iron oxide should be estimated separately by the potassium permanganate method (p 51).

Determination of Calcium Oxide.—Collect all the filtrates and wash waters from the determination of iron oxide and alumina, add sufficient hydrochloric acid to make it distinctly acid, evaporate down to a convenient volume, neutralize with ammonia, add a boiling hot solution of ammonium oxalate, and set aside for 12 hours. Collect the precipitate of calcium oxalate on a small ashless filter-paper, wash with cold water, dry, incinerate the filter-paper, ignite the whole in a weighed platinum crucible, cool, and weigh. Ignition should be continued until a constant weight is obtained. The residue is pure calcium oxide. The weight of calcium oxide ×100= per cent of calcium oxide. Per cent of calcium oxide ×1.7857= per cent of calcium carbonate.

Determination of Magnesia.—Place all the filtrates and wash waters from the determination of calcium oxide in one beaker, evaporate to small bulk, add a slight excess of ammonia, then

a solution of sodium phosphate in an excess, stir vigorously. whereupon a precipitate of ammonium magnesium phosphate will be formed. After about 15 minutes, add ammonia in an excess and set aside for several hours so that the magnesia may be completely precipitated. Collect the precipitate on an ashless filter-paper, wash with dilute ammonia (I part of ammonia with the specific gravity 0.96 and 3 parts of water) until a drop of the last filtrate acidulated with nitric acid produces no precipitate with silver nitrate. Dry the precipitate and filter-paper, incinerate the fil-

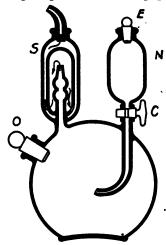


Fig. 64.—Alkalimeter.

ter-paper, ignite strongly, cool, and weigh as magnesium pyrophosphate  $(Mg_2P_2O_7)$ . The weight of the magnesium pyrophosphate multiplied by 0.36208 will give the weight of magnesium oxide. The weight of magnesium pyrophosphate multiplied by 0.7574 will give the weight of magnesium carbonate. The weight of magnesium oxide multiplied by 2.092 will give the weight of magnesium carbonate. Multiply the weight by 100 to obtain the percentage.

Determination of Carbon Dioxide.—In determining carbon dioxide in limestone, the apparatus devised by Schroetter may conveniently be used. It is called an Alkalimeter and is represented in Fig. 64. Wash the interior of the apparatus perfect-

ly clean, fill the tube S with concentrated sulphuric acid to just above the upper bulb and the tube N with dilute nitric acid nearly full, wipe the outside perfectly dry, and weigh accurately. Introduce into the bottle about 2 grams of powdered limestone through the opening O and weigh accurately again. The difference in the two weighings gives the weight of the limestone. Lift the stopper E and admit the acid into the bottle, a small quantity at a time, by opening the cock C for one second each time. The acid will decompose the limestone and carbon dioxide will pass out through the tube S in the direction indicated by the arrows. The concentrated sulphuric acid in the tube S absorbs any moisture that may vaporize with the gas and pass off otherwise. When no more carbon dioxide gas evolves, add the acid in a slight excess, replace the stopper E, heat slightly in order to expel all the carbon dioxide, cool, and weigh.

 $\frac{\text{The loss in weight} \times \text{100}}{\text{The weight of the sample taken}} = \text{per cent of carbon dioxide.}$ 

Determination of Sulphuric Anhydride.—Weigh out accurately about 5 grams of powdered limestone, dissolve in dilute hydrochloric acid, digest for a short time, dilute with water, filter, wash the residue and filter-paper thoroughly, evaporate the filtrate to a small volume, and add a solution of barium chloride, whereupon barium sulphate will be precipitated. After boiling for a few minutes, collect the precipitate on an ashless filter-paper, wash thoroughly with hot water, dry, incinerate, ignite in a platinum crucible, cool, and weigh as barium sulphate.

The weight of barium sulphate  $\times$  0.3427  $\times$  100 The weight of limestone taken

sulphuric anhydride (SO<sub>3</sub>);

The weight of barium sulphate  $\times$  0.5828  $\times$  100 = per cent of The weight of the sample taken

calcium sulphate;

per cent of calcium sulphate  $\times 0.41202$  per cent of calcium oxide. The per cent of calcium oxide calculated from the per cent of calcium sulphate should be subtracted from the per

cent of the total calcium oxide and the remainder of the calcium oxide be calculated to calcium carbonate.

Interpretation of Results of Limestone Analysis.—Good limestone usually consists mostly of calcium carbonate and small quantities of acid insoluble matter, iron oxide, and alumina.

In order to distribute carbon dioxide among the bases estimated, obtain first the per cent of calcium sulphate from the weight of barium sulphate, convert the per cent of calcium suiphate thus obtained into the per cent of calcium oxide, subtract this percentage of calcium oxide from the total per cent of calcium oxide, calculate the remainder of the total calcium oxide into calcium carbonate; subtract the difference between the percentage of calcium carbonate and calcium oxide from the percent of total carbon dioxide estimated; the remainder of the carbon dioxide should correspond to a quantity of carbon dioxide required by magnesium oxide to form magnesium carbonate.

EXAMPLE.

Assume that the following results are obtained in an analysis:

Constituents				Per cent
Calcium oxide	 	 	•••	52.355
Magnesium oxide	 	 	• • •	•54
Calcium sulphate	 • •	 		.28
Acid insoluble matter				
Iron oxide and alumina	 	 	• • •	·75
Carbon dioxide	 	 		41.65
Moisture	 • • •	 	• • •	.70

calcium oxide in calcium sulphate = 0.28×0.4102=0.115 per cent; calcium carbonate = (52.335—0.115)×1.7857=93.29 per cent; carbon dioxide required by the calcium oxide to form calcium carbonate would then be 93.29—52.24=41.05 per cent; the remainder of the carbon dioxide, that is, 41.65—41.05=0.60 per cent should correspond to the amount of carbon dioxide required by magnesium oxide to form magnesium carbonate. As the per cent of magnesium oxide is 0.54 per cent, it will require 0.54×2.092—0.54=0.59 per cent of carbon dioxide to form magnesium carbonate; hence, the per cent of magnesium carbonate = 0.54+0.59=1.13.

# FINAL STATEMENT.

IIIAD CIAILMINI.													
Constituents	Per cent												
Calcium carbonate	93.29												
Calcium sulphate	.28												
Magnesium carbonate	1.13												
Acid insoluble matter	3.68												
Iron oxide and alumina	.75												
Moisture	.70												
Undetermined	.17												
	100.00												

The determination of carbon dioxide is usually omitted as the calculation of carbonates of calcium and magnesium from their oxides is sufficiently close for technical purposes.

Analysis of Lime.—It is important to examine lime for available lime and inavailable lime in order to see whether or not a lime-kiln has been properly operated.

Determination of Calcium Oxide.—Weigh out exactly 10 grams of lime, mix with water in a mortar to form a thick milk, add a pure sugar solution of about 40° Brix in an excess, mix thoroughly so that all the lime is dissolved, forming soluble saccharate of lime, transfer the whole to a 100 c.c. flask, make up to the graduation mark with a sugar solution of the same strength, mix thoroughly, and filter. Titrate 10 c.c. of the filtrate with a standard solution of hydrochloric or sulphuric acid 1 c.c. of which is equivalent to 0.01 gram of calcium oxide, using phenolphthalein solution as indicator. The burette reading directly gives the per cent of calcium oxide. This is called available lime.

Determination of Unburned and Slaked Lime.—Slake I gram of lime with water, add 100 c.c. of the standard solution of the same strength as used for the available lime determination, heat for a few minutes, and titrate the excess of the acid with a sodium hydroxide solution of corresponding strength using phenolphthalein as indicator. 100—the number of c.c. of the sodium hydroxide solution required gives the total calcium oxide in per cent. The per cent of the total lime—the per cent of available lime gives the per cent of unburned and slaked lime or unavailable lime.

Analysis of Sulphur.—When finely divided sulphur is treated with bromine water, it becomes oxidized, the resulting product being sulphuric acid which is soluble in water while the impurities remain intact and insoluble. This principle is applied in the determination of the per cent of pure sulphur in commercial roll-sulphur.

A sample should be prepared by breaking off small pieces from a large number of rolls and by grinding them together in a mortar until no grit may be felt between the fingers. Weigh accurately about 0.5 gram of the sample, transfer to a glass-stoppered bottle of 500 c.c. capacity, add about 400 c.c. of saturated bromine water, shake vigorously, allow it to stand for one hour, shaking at intervals of a few minutes, in order to insure complete oxidation of sulphur. Transfer to a beaker, boil to expel the excess of bromine, collect the insoluble matter on a filter-paper previously dried at 100°C and weighed, wash with hot water until a drop of the last wash water forms no precipitate with barium chloride, dry at 100°C, and weight. The total weight—the weight of the filter-paper gives the weight of the impurities.

The weight of the impurity × 100
The weight of sample taken

100—per cent of impurity = per cent of pure sulphur.

Gas Analysis.—It is desirable to examine kiln gas and chimney gas for carbon dioxide, carbon monoxide, and oxygen in order to control the kiln and boiler-house properly. Fig. 65 represents the apparatus employed for the determination of these gases. It is known as Orsat's Gas Apparatus. It consists of a burette A for measuring gases and U-tubes B, C, D. The burette is of 100 c.c. capacity, graduated in tenths, and provided with a water-jacket. All the U-tubes are filled with small glass tubes so that the gases to be absorbed will be exposed to a large surface. The small glass tubes in the U-tube D are filled with small copper rods. Communications between the burette and the U-tubes are closed or opened by means of the cocks b, c, d, and e is a three-way cock. When the handle of the cock e is at right

angles with the tube F, the burette opens a communication with the gas-pipe or chimney through the tube f. When the handle is parallel to F, the burette opens communication with the atmosphere through the tube g. When turned one-eighth, it cuts both communications. The bottle E is filled with water, con-

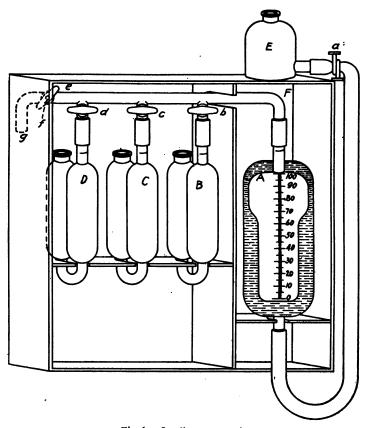


Fig. 65.—Orsat's gas apparatus.

nected with the lower end of the burette by means of a rubber tubing. When lowered, it draws gas into the burette; on the other hand, when raised, it forces gas out of the burette. B is filled with 110 c.c. of potassium hydroxide solution of specific gravity 1.26 and is used for absorbing carbon dioxide. C is filled

with 18 grams of pyrogallic acid dissolved in 40 c.c. of hot water and 70 c.c. of potassium hydroxide solution of specific gravity 1.26. D is filled with 110 c.c. of a solution of cuprous chloride. Cuprous chloride may be prepared as follows: dissolve 35 grams cupric chloride in 200 c.c. of concentrated hydrochloric acid, transfer to a glass-stoppered bottle, add a small piece of metallic copper, set aside for two days, shaking occasionally, then add 120 c.c. of water. In order to prepare the apparatus suitable for gas analysis, close the cocks b, c, d, open the burette to the atmosphere by turning the three-way cock handle parallel to the tube F, elevate the bottle E, keeping the pinch-cock a open until the water begins to run out of the tube q, then close the pinch-cock a and the three-way cock e. Now open the cock b and lower the bottle E, keeping the pinch-cock a open until the liquid in B reaches the mark, then close a and b. The liquids in C and D may be brought up to the marks in the same manner. The apparatus must be brought to this condition always before it is to be used for analysis.

Direction for Examination of Kiln Gas.—Open the burette to the tube f by turning the three-way cock e square to the tube F, lift the bottle E, keeping the pinch-cock a open until the water begins to run out of the tube f, then close a, connect the tube f with the gas-pipe by means of a rubber tubing, and open a to allow the gas to fill the burette. When this is full, close a, and disconnect f from the gas-pipe. Hold the bottle E at such a height that the surface of the water therein comes to a level with the zero mark of the burette, bring the level of the water within the burette exactly to the zero mark by cautiously opening the pinchcock a, then close the three-way cock e, by turning it one-eighth. Open the cock b, elevate the bottle E, keeping the pinch-cock aopen until the burette is nearly filled with water, then lower E until the liquid within B nearly reaches the mark. Care should be taken that the liquid in B never passes the mark. Repeat this operation several times to insure complete absorption of carbon dioxide. Now allow the liquid in B to come up exactly to the mark, close b, hold the bottle E at such a height that the surface of the water therein comes exactly to a level with that of the water in the burette, then close a, and take the reading. Repeat the absorption and reading until a constant reading is obtained. This reading gives the volume per cent of carbon dioxide in the gas. The tubes C and D may be operated successively exactly in the same manner. C absorbs oxygen, D, carbon monoxide. The reading after the absorption by C minus the reading after the absorption by B gives the volume per cent of oxygen; the reading after the absorption by C gives the volume per cent of carbon monoxide.

Frequently, traces of hydrogen sulphide (H<sub>2</sub>S) and sulphurous acid are found in kiln gas. The former is detrimental to beetjuice as it combines with iron, forming black sulphide of iron and imparting a dark color to juice or syrups. It may be detected by passing the gas into a solution of lead acetate when a black precipitate of lead sulphide will be formed if hydrogen sulphide is present.

The Examination of Chimney Gas is conducted exactly in the same manner as that of kiln-gas. The gas may be drawn from the chimney into the burette in the following manner: after arranging the apparatus for analysis, connect the tube f with a long glass or metallic tubing inserted into the chimney by means of rubber tubing, set the three-way cock e at right angles to the tube F, so that the burette will be in communication with the interior of the chimney, lower the bottle E, keeping the pinch-cock e0 open, whereupon the gas will be drawn into the burette. When the burette is filled with gas, close e0, turn e1 parallel to e1, so that the burette will be open to the atmosphere, then lift the bottle e2, so that the gas will be forced out of the burette. Repeat several times before a sample of gas for analysis is obtained, in order to make it sure that a proper sample is taken. Then proceed in the analysis.

Analysis of Coke.—Success in making good gas and well burned lime more or less depends upon the quality of the coke. It is very important to select coke that contains the smallest possible quantities of ash and sulphur. High ash content tends to introduce undesirable impurities into lime, such as iron and silica;

high sulphur content is liable to form hydrogen sulphide which is detrimental to juice. It is a common practice, for this reason, to examine coke for ash and sulphur.

Sampling.—Break off small pieces from a large number of lumps of coke, reduce these pieces to the size of peas, mix thoroughly, and obtain a small sample by quartering and requartering. Grind the sample thus obtained until all pass through a 80-mesh sieve and keep in a glass-stoppered bottle for analysis.

Determination of Moisture.—Weigh out accurately about 3 grams of the sample in a weighed weighing bottle or watch-glass, dry in an air oven heated to 110°C for about 3 hours, or to constant weight.

The loss in weight  $\times$  100 The weight of coke taken = per cent of moisture.

Determination of Ash.—Weigh out accurately about 2 grams of the sample in a weighed platinum dish, burn first the easily combustible matter at a low temperature, then heat at high temperature until all the carbonaceous matter is consumed or a constant weight is obtained, cool, and weigh.

The weight of the residue  $\times$  100 The weight of coke taken = per cent of ash.

Determination of Sulphur.—Weigh out accurately I gram of the sample, mix with 10 grams anhydrous sodium carbonate and 5 grams potassium nitrate very intimately on a glazed paper. Heat a platinum crucible of 50 c.c. capacity to a dull redness, introduce a small portion of the mixture into the crucible and allow it to deflagrate, whereupon the oxidation of carbon and sulphur will readily take place. When combustion is complete, add a small portion of the mixture again and allow it to deflagrate. It is advisable to use alcohol or gasoline burner instead of gas for this work, to avoid contamination with sulphur from gas. Continue this operation until all the mixture is transferred to the crucible and heat at a still higher temperature for 10 minutes to insure that all the carbonaceous matter is completely consumed, then cool, dissolve the fused mass in hot water, filter, and wash the residue and filter-paper thoroughly with hot water. Now

collect all the filtrate and wash water in one beaker, acidify with hydrochloric acid, and add an excess of a solution of barium chloride, whereupon barium sulphate, which is insoluble in water and acid, will be precipitated. Allow it to stand for 12 hours, filter, wash the precipitate and filter-paper thoroughly with hot water, dry, incinerate, ignite, cool, and weigh as barium sulphate.

The weight of barium sulphate  $\times$  0.13734  $\times$  100 = Per cent of Sulphur.

Analysis of Coal.—Moisture, ash, and sulphur in coal are determined exactly in the same manner as those in coke.

As to the determination of volatile matter, fixed carbon, and the caloric power of coal and coke, the readers are referred to Stillman's Engineering Chemistry.

Water Analysis.—As the importance of the examination of water for sugar-house use has been discussed under WATER, only the method for analysis will be described in this article.

Sampling.—Wash a glass-stoppered bottle of one gallon capacity with hydrochloric acid, then with water, and finally rinse with the water to be sampled. If the sample is to be taken from a stream, fill the bottle with a small portion at a time at intervals of a few minutes until the bottle is full. If the sample is to be taken from a well, pump out water for a few minutes and then fill the bottle for sample.

Determination of Suspended Matter.—Water sometimes contains suspended matter; it is desirable to estimate the quantity thereof before proceeding in the determination of soluble matter. Shake the water in the sample bottle well, measure out exactly 1000 c.c. in a graduated flask, filter through a quantitative filter-paper, previously dried and weighed accurately, wash the residue and the filter-paper thoroughly with hot distilled water, dry to constant weight at 100°C, and weigh. (The final weight—the weight of the filter-paper) ×100= the suspended matter in parts per 100,000.

Determination of Total Soluble Matter.—Clean a platinum evaporator thoroughly, ignite, and weigh accurately, then evapo-

rate all the filtrate obtained from the determination of suspended matter on a water bath. After the evaporation is completed, heat the dish and contents in an air oven at 105°C for 30 minutes, cool, and weigh. (The weight of the evaporator and the contents—the weight of the evaporator) ×100= the total soluble matter in parts per 100,000.

Determination of Organic and Other Volatile Matter.—Ignite the total solid matter just obtained at a dull red heat to constant weight, cool, and weigh. The loss in weight ×100= the organic and other volatile matter in parts per 100,000.

Determination of Acid Insoluble Matter.—Dissolve the total solid matter just obtained in a few c.c. of hydrochloric acid, evaporate to perfect dryness on a hot water bath, then heat in an air oven at 110°C for one-half hour, moisten with a few drops of concentrated hydrochloric acid, and continue heating in the air oven at the same temperature for one hour. Now treat the contents of the dish with 10 c.c. hydrochloric acid and 50 c.c of water, boil, filter through an ashless filter-paper, wash the residue in the filter-paper with hot water until a drop of the last wash water forms no precipitate with silver nitrate. Dry the residue and filter-paper, incinerate, ignite in a weighed platinum crucible, cool, and weigh. The weight of the residue ×100= the acid insoluble matter in parts per 100,000.

Collect all the filtrate and wash water from the determination of the acid insoluble matter in one beaker, evaporate down to a small volume, transfer to a 100 c.c. flask, and then complete the volume to the graduation mark with water. Use 25 c.c. of this solution for the determination of sulphuric anhydride and the remaining 75 c.c. for the determination of iron oxide, alumina, lime, and so forth.

Determination of Sulphuric Anhydride ( $SO_3$ ).—Add an excess of barium chloride solution to 25 c.c. of the solution just assigned, whereupon sulphuric acid will be precipitated as barium sulphate. Filter the precipitate, wash, dry, ignite, and weigh. The weight of the barium sulphate  $\times \frac{100}{25} \times 0.34293 \times 100 = \text{anhydrous sulphuric acid } (SO_3)$  in parts per 100,000.

Determination of Iron Oxide and Alumina.—Determine iron oxide and alumina in 75 c.c. of the solution just assigned in the same manner as those in limestone. The weight of the iron oxide and alumina  $\times \frac{100}{75} \times 100 = \text{iron oxide}$  and alumina in parts per 100,000.

Determination of Calcium Oxide.—Determine calcium oxide in the filtrate obtained from the determination of iron oxide and alumina in the same manner as that in limestone. The weight of calcium oxide  $\times \frac{100}{75} \times 100 = \text{calcium oxide in parts per}$  100,000.

Collective Determination of Magnesia, Potash, and Soda as Sulphates.—Collect all the filtrate and wash water from the determination of calcium oxide in one beaker, boil down to a small volume, transfer, after filtering if turbid, to a weighed platinum dish, acidify with sulphuric acid, evaporate to perfect dryness, ignite, cool, and weigh. The residue consists of sulphates of magnesium, potassium, and sodium. Note the weight of this residue so that the weight of sodium sulphate may be obtained after determining the weights of sulphates of magnesium and potassium.

It is a common practice to estimate magnesia as magnesium pyrophosphate and potash as potassium platinum chloride. The magnesium pyrophosphate and potassium platinum chloride are then converted into sulphates of the respective bases by calculation. Sodium sulphate is then obtained by subtracting sulphates of magnesium and potassium from the result of the collective determination. In order to carry out this plan, the sulphates obtained from the collective determination are dissolved in water, transferred to a 100 c.c. flask, and made up to the graduation mark with water, and mixed thoroughly. One-half of this solution (50 c.c.) is used for determination of magnesia and the other half for that of potash.

Determination of Magnesia.—To 50 c.c. of the solution just assigned, add a few drops of hydrochloric acid, an excess of am-

100,000.

monia and then a solution of disodium phosphate, stirring constantly until no more precipitate forms, and allow it to stand for several hours. Collect the precipitate on an ashless filter-paper, wash, dry, ignite, and weigh as magnesium pyrophosphate ( $Mg_2P_2O_7$ ). The weight of magnesium pyrophosphate  $\times 1.081$  the weight of magnesium sulphate. The weight of magnesium

pyrophosphate  $\times \frac{100}{37.5} \times 0.3624 \times 100 =$  the magnesia in parts per 100,000.

Determination of Potash.—To the other 50 c.c. of the solution assigned, add a few drops of hydrochloric acid and a slight excess of platinum chloride (PtCl<sub>4</sub>), evaporate down to a syrupy condition, then add 20 c.c. of 80 per cent alcohol, whereupon, since it is insoluble in alcohol, potassium platinum chloride ( $K_2$ PtCl<sub>6</sub>) will be precipitated. Collect the precipitate on a filter-paper previously dried and weighed, wash with 80 per cent alcohol until the last filtrate is perfectly clear, dry, and weigh. The weight of potassium platinum chloride  $\times 0.3587$ = the weight of potassium sulphate. The weight of potassium platinum chloride  $\times 100$   $\times 100$   $\times 100$  = the potash in parts per

num chloride  $\times \frac{100}{37.5} \times 0.1939 \times 100 =$  the potash in parts per 100,000.

Soda.—The weight of sodium sulphate may be obtained by subtracting twice the weights of sulphates of magnesium and potassium from the weight of sulphates of magnesium, potassium, and sodium obtained in the collective determination. The weight of sodium sulphate $\times$ 0.4368 $\times \frac{100}{75.0} \times 100 =$  the soda in parts per

Determination of Chlorine.—Boil down 100 c.c. of the sample of water to small volume, add 2 c.c. of concentrated nitric acid, filter if turbid, heat to boiling, then add a solution of silver nitrate, a little at a time, stirring constantly until no more precipitate forms, continue boiling until the precipitate assumes a granular appearance and the solution becomes almost clear. Now filter through an ashless filter-paper, wash the precipitate first

with hot water acidified with nitric acid until the last wash water forms no precipitate with hydrochloric acid, then with about 50 c.c. of cold water. Dry the precipitate and filter-paper in an air oven at 120° C until perfectly dry, transfer the precipitate to a small porcelain crucible, previously weighed, as much as possible by scraping the filter-paper with a fine platinum wire, hold the filter-paper with the platinum wire over the crucible and incinerate it completely, allowing any incombustible portion to fall into the crucible. Moisten the contents of the crucible with nitric acid and after a few minutes with hydrochloric acid, in order to convert any reduced silver into silver chloride. Heat the crucible very cautiously over a small direct flame, in order to evaporate the acids, until perfectly dry, then raise the temperature slightly, and continue heating until the silver chloride fuses, cool, and weigh. The weight of silver chloride  $\times 0.24726 \times 1000 =$ the chlorine in parts per 100,000.

The result of the analysis may be stated in the manner shown in the following example:

### WATER ANALYSIS.

Substances	Parts per 100,000
Acid insoluble matter	
Iron oxide and alumina	
Chlorine (Cl)	
Sulphuric anhydride (SO <sub>3</sub> )	3.11
Sodium oxide (Na <sub>2</sub> O)	
Potassium oxide (K <sub>2</sub> O)	
Calcium oxide (CaO)	2.24
Magnesium oxide (MgO)	
Volatile matter	4.45
	14.240
Oxygen in excess of chlorine	
Total solid matter	14.095
Volatile matter includes carbon dioxide, organic forth.	c matter, and so
torer	

Oxygen in excess of chlorine may be found by the formula,  $\frac{O}{2Cl}$ 

$$\times$$
 parts of chlorine; for example,  $\frac{16}{2\times35.45}$   $\times$  0.64 = 0.145.

Interpretation of Analytical Results.—It is customary to unite acids and bases, as they may exist in chemical combination in nature under ordinary circumstances. Chlorine is usually combined with sodium, if still in excess, then with potassium, magnesium, and finally with calcium; sulphuric acid with sodium, potassium, calcium, and finally with magnesium; carbon dioxide with calcium and magnesium. The method of calculation may be best shown by examples, using the values stated in the preceding article.

## EXAMPLE.

Sodium Chloride.—The parts of sodium oxide 0.64 parts of chlorine require to form sodium chloride may be found by the following proportion:

70.9 (Cl<sub>1</sub>): 62.0 (Na<sub>2</sub>O):: 0.64: parts of sodium oxide; therefore, the parts of sodium oxide combined with 0.64 parts of chlorine =  $\frac{62 \times 0.64}{70.9}$  = 0.56 parts; and the parts of sodium in 0.56 parts of sodium oxide may be found by the following proportion: 62.0 (Na<sub>2</sub>O): 46.0 (Na<sub>2</sub>):: 0.56: parts of sodium; therefore the parts of sodium =  $\frac{0.56 \times 46}{62}$  = 0.4155. This value indicates the parts of sodium combined with 0.64 parts of chlorine, then, 0.64 + 0.415 = 1.055 = sodium chloride in parts per 100,000.

Sodium Sulphate.—The parts of sulphuric anhydride 0.78—0.56=0.22 parts of sodium oxide require to form sodium sulphate may be found by the following proportion:

62.0 (Na,O): 80.0 (SO<sub>8</sub>):: 0.22: parts of sulphuric anhydride; hence, the parts of sulphuric anhydride combined with 0.22 parts of sodium oxide  $=\frac{0.22\times80}{62}$  = 0.284; then, 0.22 + 0.284 = 0.504 = sodium sulphate in parts per 100,000.

Potassium Sulphate.—The parts of sulphuric anhydride 0.48 parts of potassium oxide require to form potassium sulphate may be found by the following proportion:

94.0 (K<sub>2</sub>O): 80.0 (SO<sub>3</sub>):: 0.48: parts of sulphuric anhydride;

hence, the parts of sulphuric anhydride combined with 0.48 parts of potassium oxide  $=\frac{0.48 \times 80}{94} = 0.408$ ; then, 0.48 + 0.408 = 0.888 = potassium sulphate in parts per 100,000.

There still remain 3.11—0.284—0.408=2.418 parts of sulphuric anhydride uncombined.

Calcium Sulphate.—The parts of calcium oxide 2.418 parts of sulphuric anhydride require to form calcium sulphate may be found by the following proportion:

80.0 (SO<sub>3</sub>): 56.0 (CaO) :: 2.418 : parts of calcium oxide; therefore, the parts of calcium oxide combined with 2.418 parts of sulphuric anhydride =  $\frac{2.418 \times 56}{80}$  =1.693; then, 2.418 + 1.693 = 4.111 = calcium sulphate in parts per 100,000.

There still remain 2.24—1.693=0.547 parts of calcium oxide uncombined.

Calcium Carbonate.—The parts of carbon dioxide (volatile matter) 0.547 parts of calcium oxide require to form calcium carbonate may be found by the following proportion:

56.'o (CaO): 44.0 (CO<sub>3</sub>)::0.547: parts of carbon dioxide; hence the parts of carbon dioxide combined with 0.547 parts of calcium oxide =  $\frac{0.547 \times 44}{56}$  = 0.430; then, 0.547 + 0.430 = 0.977 = calcium carbonate in parts per 100,000.

0.430 parts should be subtracted from the parts of volatile matter as parts of carbon dioxide combined with calcium oxide.

Magnesium Carbonate.—The parts of carbon dioxide 0.98 parts of magnesium oxide require to form magnesium carbonate may be found by the following proportion:

40.0 (MgO): 44.0 (CO<sub>2</sub>):: 0.98: parts of carbon dioxide; hence, the parts of carbon dioxide combined with 0.98 parts of magnesium oxide =  $\frac{0.98 \times 44}{40}$  = 1.078; then, 0.98 + 1.078 = 2.058 = magnesium carbonate in parts per 100,000.

1.078 parts should also be subtracted from the parts of volatile

matter as parts of carbon dioxide combined with magnesium oxide.

There still remain 4.45—0.430—1.078=2.942 parts of volatile matter. This should be recorded as organic and other volatile matter.

The result may be stated as follows:

Substances	Parts per 100,000
Acid insoluble matter	900
Iron oxide and alumina	
Sodium chloride	1.055
Sodium sulphate	504
Potassium sulphate	
Calcium sulphate	4.111
Calcium carbonate	977
Magnesium carbonate	2.058
Volatile matter	2.642
Total solid matter	14.095

In order to obtain the results in grains per U. S. gallon, the parts per 100,000 must be multiplied by 0.583.

Stock Samples.—At the end of a period or campaign, it is necessary for a sugar-house management to know how much granulated sugar has been manufactured and how much crystallizable sugar in syrups there is in stock, in order to compute the loss of sugar in the process of manufacturing.

In order to calculate the amount of crystallizable sugar in syrups in stock, 3 data are required:—the weight, the Brix, and the purity of syrups. It is customary to obtain the weight of syrups in kilograms. Quantity of syrups are first obtained in hectoliters, as the capacities of tanks and other storage vessels in sugar-houses are usually calculated in hectoliters. Most sugar-houses are provided with lists of tanks and other vessels giving their capacities in hectoliters and heights in centimeters. The volume of syrups in the tanks and other vessels may be readily obtained by simple calculation, after finding the number of centimeters of the vessel that are out above the syrup. The numbers of hectoliters of syrups are converted into those of kilograms by multiplying by 100 and by the specific gravity of the syrup, since one hectoliter contains 100 liters and the specific

gravity of syrup represents approximately the weight of one liter of the syrup in kilograms.

The Brix and purity of the undiluted syrups in stock are determined the same as those of fillmass (p. 239) and the specific gravity of undiluted syrups may be found directly above the Brix of undiluted syrups in Table IV. The weight of wet syrups is converted into that of dry substance by multiplying by the Brix degree. As stated previously, the dry substance or Brix and the purity obtained by this method are apparent, not true. If exact dry substance and purity are desired, determination of water in each syrup should be made by drying as directed under Determination of Water and Total Solids in Syrups and Fillmasses.

After the dry substance and purity have been obtained, crystallizable sugar may be calculated by Dupont's formula for crystallized sugar with a slight modification in data (p. 270),

Crystallizable sugar = 
$$\frac{a-P'}{P-P'}$$

in which a=the purity of dry substance of syrup, P'=the purity of dry substance of molasses, P= the purity of granulated sugar, —usually assumed as 100.

Example: Assume a = 78.0, P' = 60.0, P = 100, weight of dry substance of syrup = 158,679 kg.; then,

crystallizable sugar = 
$$\frac{78.0-60.0}{100-60.0} \times 158,679 = 71,405 \text{ kg}.$$

158,679 - 71, 405 = 87,274 kg. = the dry substance of molasses from which sugar could not be recovered by the process of crystallization.

When various quantities of syrups of various purity are in stock, the average purity of the total weight of syrups must be obtained from calculation. The total weight of sugar in kilograms is obtained by multiplying the number of kilograms of each syrup by their respective purity and adding their products. The average purity is obtained by dividing the total weight of the sugar by the total weight of the dry substances and multiplying by 100. This may be best shown by example:

Weight of syrups in kilograms	Brix	Weight of dry sub- stance in kilograms	Purity	Weight of sugar in kilograms
48,356	85.4	41,296	74.5	30,765
60,984	78.5	47,872	82.4	39,446
176,245	80.6	142,053	66.2	94,039
Total weight		231,221		164,250

Average purity 
$$=\frac{164,250\times100}{231,221} = 71.04$$
; then, crystallizable sugar  $= 231,221 \times \frac{71.04-60}{100-60} = 63,817$  kg.; dry substance of molasses  $= 231,221-63,817 = 167,404$  kg.

The percentage of sugar in molasses that may be recovered varies according to the process employed for extracting sugar therefrom. It is claimed that Steffen's process recovers 66 per cent of sugar in molasses; hence, assuming that 60 per cent of the dry substance of molasses is sugar, the weight of the dry substance of molasses  $\times 0.60 \times 0.66$  would be the weight of sugar restorable from molasses. Having 167,404 kg. of dry substance of molasses in stock, restorable sugar by Steffen's process would be  $167,404 \times 0.60 \times 0.66 = 66,292$  kg. Then the total sugar that may be recovered from 231,221 kg. of dry substance of syrups in a sugar-house where Steffen's process is in use would be 63.817 + 66,292 = 130,109 kg.

The factor for obtaining the weight of recoverable sugar from molasses by the osmose process is variable, according to the quality of molasses to be osmosed and the nature of the water to be used for the process, and also, according to the number of times the molasses is worked through the process. Generally, however, every time molasses is worked through the process once, 12 per cent of the weight of the initial molasses is returned as crystallized sugar, 65 per cent of the same is returned as molasses. Molasses is worked through the osmose process in some factories once, in some, twice, in some, three times. Therefore, the factor for obtaining crystallizable sugar from molasses by the osmose process should be established according to the number of times the molasses is worked through the process.

Examination of Beet-Seeds.—Beet-seeds are usually examined for moisture, per cent of foreign matter, number of seed-balls per kilogram or pound, and germinating power.

The requirements for acceptable seeds are as follows: (1) moisture should not exceed 14 per cent, (2) foreign matter should not exceed 3 per cent, (3) 75 per cent seed-balls should germinate in 15 days, (4) 1 kilogram of seeds should produce not less than 70,000 sprouts; of this number, 46,000 should appear within 6 days.

Sampling.—A sample should be taken in such a manner as to represent an entire lot of seeds by taking a small quantity from each of a large number of bags. The simplest instrument for taking a small sample from beet-seed bags is a metallic tube provided with a solid point at one end, a handle at the other and with a side opening for receiving sample of seeds. Boring such a tube into a seed bag, the seeds will fill the tube through the side opening and be taken out for a sample. After taking samples from a large number of bags, mix thoroughly, obtain smaller sample for testing by quartering and requartering.

Determination of Moisture.—Weigh out exactly 10 grams of the sample in a glass-stoppered weighing bottle, dry at 105°C in an air oven to constant weight.

The loss in weight  $\times$  100 = per cent of moisture.

Determination of Foreign Matter.—After mixing the whole sample thoroughly, weigh out 50 grams, clean each seed-ball by holding in a pair of forceps and brushing. When all the seeds are carefully cleaned, weigh them again.

 $\frac{(50 - \text{the final weight}) \times 100}{50} = \text{per cent of foreign matter};$ 100—per cent of foreign matter = per cent of pure seeds.

Number of Seeds per Kilogram or per Pound.—Count the clean seeds obtained from the determination of foreign matter. Denote the number obtained by a; then,

 $a \times \frac{1000}{50}$  = number of seeds per kilogram;

 $\frac{\text{Number of seeds per kg.}}{\text{number of seeds per pound.}} = \text{number of seeds per pound.}$ 

Determination of Germinating Power.—Count out 100 seeds from a thoroughly mixed sample, place them between the folds of a thick blotting paper, and keep moist constantly. The temperature of the chamber in which germination is conducted should be kept at 20°C (68°F) 18 hours out of each 24 hours. Never allow the temperature to fall below 15°C (59°F) or rise above 32°C (80.6°F). As soon as the seeds begin to germinate, count the number of seed-balls germinated and the number of sprouts everyday. Place the seed-balls that have the same number of sprouts in one group. Should any seed-balls have more sprouts when counted the succeeding day than the preceding day, transfer them to the proper groups. The result of counting should be recorded every day. A period of two weeks is the usual length of time required for the germination test. When the germination test is completed, the following statements should be made:

- 1. Number of seed-balls germinated with certain number of sprouts per 100 seed-balls, as, for example, 10 seed-balls germinated with 2 sprouts, 20 seed-balls germinated with 3 sprouts, and so forth.
  - 2. Total number of seed-balls germinated per 100 seed-balls.
  - 3. Total number of sprouts per 100 seed-balls.
  - 4. Total number of sprouts per kilogram or pound.

# APPENDIX.

# TABLE I.

Corrections of the Brix Degree for Variations in Temperature from the Normal, 17.5°C (63.5°F), prepared by Sachs. Page 297.

# TABLE II.

Comparison of the Degrees Brix and Baumé and of the Specific Gravity of Sugar Solutions at 17.5°C, prepared by Stammer. Page 298.

# TABLE III.

Per cent of Sucrose calculated for the 110 c.c. Volume Method, prepared by Schmitz. (See page 172 for the directions for use). Pages 299-302.

### TABLE IV.

Per cent of Sucrose calculated for the 110 c.c. Volume Method and Purity of Dilute Juices (7.1° to 27.0° Brix) and Brix of undiluted thick syrups, prepared by Nikaido. (See pages 172, 239 for directions for use). P. R. = polariscope reading. Sug. = per cent of sugar. Sp. gr. = specific gravity. Und. syp. = undiluted syrup. Pages 303-339.

# TABLE V.

Per cent of Sucrose calculated for the 110 c.c. Volume Method and Purity of Osmose Waste Water, prepared by Nikaido. Page 340.

# TABLE VI.

Amount of Calcium Oxide in Milk of Lime of various densities at 15°C, prepared by Blatner. Page 341.



TABLE I.

Temp.	Temp.				App	roxima	ate Br	ix De	угее					
C.	F.	0	5.	10	15	20	25	, 30	35	40	50	60	70	75
						SUBT	RACT							
0	32	.27	.30	.41	.52	.62	.72	.82	.92	.98	1.11	1.22	1.25	1.29
5	41	.23	-30	.37	.44	.52	-59	.65	.72	-75	.80	.88	.91	-94
10	50	.20	.26	.20	-33	.36	.39	.42	.45	.48	.50	.54	.58	.61
11	51.8	.18	.23	.26	.28	.31	.34	.36	-39	.41	.43	.47	.50	-53
12	53.6	.16	.20	.22	.24	.26	.29	.31	•33	.34	.36	.40	.42	.46
13	55.4	.14	.18	.19	.21	.22	.24	.26	.27	.28	.29	-33	-35	-39
14	57-2	.12	.15	.16	.17	.18	.19	.21	.22	.22	.23	.26	.28	.32
15	59	.09	.11	.12	.14	.14	.15	.16	.17	.16	.17	.19	.21	.25
16	60.8	.06	.07	.08	.09	.10	.10	.11	.12	.12	.12	.14	.16	.18
17	62.6	.02	.02	.03	.03	.03	.04	.04	.04	.04	.04	.05	.05	.06
						Al	D <b>D</b>							
18	64.4	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.02
19	66.2	06	.08	.08	.09	.09	.10	.10	.10	.10	.Io	.10	.08	.06
20	68.o	.11	.14	.15	.17	.17	.18	.18	.18	.19	.19	.18	.15	.II
21	69.8	.16	.20	.22	.24	.24	.25	.25	.25	.26	.26	.25	.22	81.
22	71.6	.21	.26	.29	.31	.31	.32	.32	.32	-33	•34	.32	.29	.25
23	73.4	.27	.32	-35	.37	38ء	.39	.39	.39	.40	.42	.39	.36	-33
24	75.2	.32	.38	.41	.43	-44	.46	.46	-47	-47	.50	.46	•43	.40
25	77.0	.37	.44	-47	.49	.51	- ∙53	•54	-55	∙55	.58	·5 <b>4</b>	.51	.48
26	78.8	-43	.50	-54	.56	.58	.60	.61	.62	.62	.66	.62	.58	-55
27	80.6	-49	-57	.61	.63	.65	.68	.68	.69	.70	.74	.70	.65	.62
28	82.4	.56	.64	.68	.70	.72	.76	.76	.78	.78	.82	.78	.72	.70
29	84.2	.63	.7 I	·75	.78	-79	.84	.84	.86	.86	.90	.86	.80	.78
30	86	.70	.78	.82	.87	.87	.92	.92	٠94	.94	.98	•94	.88	.86
35	95	1.10	1.17	1,22	1.24	1.30	1.32	1.33	1.35	1.36		1.34		1.25
40	104	1.50	1.61	1.67	1.71	1.73	1.79	1.79	1.80	1.82			1.69	
50	122		2.65	2.71	2.74	2.78	2.80	2.80	2.80	2.80			2.56	
60	140		3.87	3.88	3.88	3.88	3.88	3.88	3.88	3.90	-		3-43	
70	158		5.17	5.18	5.20	5.14	5.13	5.10	5.08	5.06			4-47	
80	176			6.62	6.59	6.54	6.46	6.38	6.30	6.26		-	5.50	
90	194			8.26	8.16	8.06	7.97	7.83	7.71	7.58	7.30	6.96	6.58	6.37

TABLE II.

Brix degree .1°Bx = .056°B <b>é</b>	.1°Bé = .177°Bx	Specific gravity .coc48 = .1°Bx .coc84 = .1°B&	Brix degree .1°Bx == .054°B <b>é</b>	Baumé degree .1°Bé = .185°Bx	Specific gravity .coo64 = .1°Bx .co131 = .1°Bé
10	5. <i>7</i>				
11	6.2	1.0401	53	29.3	1.2495
12	6.8	1.0443 1.0485	54	29.8	1.2552
13	7.4	1.0527	55	30.4	1.2608 1.2666
14	7.9	1.0570	56	30.9	1.2000
15	8.5	1.0613	57 58	31.4 31.9	1.2723
16	9.0	1.0656	59	32.5	1.2840
17	9.6	1.0700	60	33.0	1.2899
18	10.1	1.0744	61	33.5	1.2958
19	10.7	1.0788	62	34.0	. 1.3018
20	11.3	1.0833	63	34.5	1.3078
21	11.8	1.0878	64	35.1	1.3138
22	12.4	1.0923	65	35.6	1.3199
23	13.0	1.0968	66	36.1	1.3260
24	13.5	1.1014	67	36.6	1.3322
25	14.1	1.1061	68	37.1	1.3384
26	14.6	1.1107	69	37.6	1.3446
27	15.2	1.1154	70	38.1	1.3509
28	15.7	1.1201	71	38.6	1.3572
29	16.3	1.1249	72	39.1	1.3635
30	16.8	1.1297	73	39.6	1.3699
31	17.4	1.1345	74	40.1	1.3764
32	17.9	1.1393	75	40.6	1.3829
33	18.5	1.1442	76	41.1	1.3894
34	19.0	1.1491	77	41.6	1.3959
35	• 19.6	1.1541	78	42.1	1.4025
36	20.1	1.1592	79	42.6	1.4092
37	20.7	1.1641	8o	43.I	1.4158
38	21.2	1.1692	81	43.6	1.4226
39	21.8	1.1743	82	44.I	1.4293
40	22.3	1.1794	83	44.6	1.4361
41	22.9	1.1846	84	45.I	1.4430
42	23.4	1.1898	85	45.5	1.4498
43	23.9	1.1950	86	46.0	1.4568
44	24.5	1.2003	87	46.5	1.4637
45	25.0	1.2056	88	47.0	1.4707
46	25.6	1.2110	89	47.4	1.4778
47	26.1	1.2164	90	47.9	1.4848
48	26.6	1.2218	91	48.4	1.4920
49	27.2	1.2273	92	48.9	1.4991
50	27.7	1.2328	93	49.4	1.5063
51	28.2	1.2383	94	49.8	1.5136
52	28.8	1.2439	95	50.3	1.5209

### **TABLES**

0.0 9.5 8.5. 6.0 0. 28.8 1 25 2 4 4 82. 57. 57. 1.13. 58. 59. 69. 82. 72. 83. 84. 89. 89.

25 25 25 25

1.5

0.1

ŵ 6

# TABLE III.—(Continued.)

 $\begin{array}{c} ... \\$ 16.5 14.0  $\begin{array}{c} 4.38 \\ 2.$ 13.5 12.5 7.56.

		P.R.	9	4	43	£	4	45	9	44	<del>&amp;</del>	<b>6</b>	S	5	2	53	2	55	2	22	85 80	20	٤,	55	35	•
	17.5		10.67	10.01	11.20	11.47	11.74	12.01	12.27	12.54	12.81	13.07	13.34	13.61	13.88	14.14	14.41	14.68	14.94	15.21	15.48	15.75	16.01	10.25	16.83	
	17.0		10.69	10.96	11.23	11.49	92.11	12.03	12.30	12.56	12.83	13.10	13.37	13.64	13.90	14.17	14.44	14.71	14.97	15.24	15.51	15.78	16.05	10.31		
	16.5		10.71	10.98	11.25	11.52	11.79	12.05	12.32	12.59	12.86	13.13	13.40	13.66	13.93	14.20	14.47	14.74	15.00	15.27	15.54	18.81				
	16.0		10.73	11.00	11.27	11.54	18.11	12.08	12.35	12.61	12.88	13.15	13.42	13.69	13.96	14.23	14.50	14-77	15.03	15.30	15.57					
	15.5		10.76	11.03	11.29	11.56	11.83	12.10	12.37	12.64	12.91	13.18	13.45	13.72	13.99	14.26	14.53	14.80								
TABLE III.—(Continued.) Brix	15.0	rat	10.78	11.05	11.32	11.59	11.86	12.13	12.40	12.67	12.94	13.21	13.48	13.75	14.02	14.29										
III.—(C Brix	14.5	cent. Sugar	10.80	11.07	11.34	11.61	11.88	12.15	12.42	12.69	12.97	13.23	13.50	13.78			•									
TABLE	14.0	Per	10.82	11.09	11.36	11.64	16.11	12.18	12.45	12.72	12.99	13.26														
	13.5		10.84	11.12	11.39	99.11	11.93	12.20	12.47	12.74	13.02	ı														
	13.0		10.86	11.14	11.41	11.68	11.95	12.23	12.50						24 - 24 Be	404	દ્રં	ķ,	8 8	2:	?	9.9	12.	23		
	12.5		10.89	91.11	11.43	11.71	11.98	12.25						Perc. sug.	40.50		દ	Š	8 :	1;	24	9 9	21	7.		
	12.0		16.01	11.18	11.46									.,	:	11:3	•	•	•	•	•	•	• ``	•	-	
	11.5		10.93										L	P.R		_	۳.	i.		4,	٠٠			٠		
		د.																								

		P.R.		<del>\$</del> ;	4 4	43	4	. <del>4</del>	<b>4</b>	47	84	49	20	54	5.2	53	54	55	26	22	20 20 20	29	9	9	62	63	9	59	9	62	89	69	2	17	72	73	74	75	92	11	28	79	õ
	24.0		,	10.33	10.00	11.12	11.42	11.69	11.94	12.21	12.47	12.73	12.99	13.25	13.51	13.77	14.02	14.29	14.55	14.81	15.07	15.33	15.59	15.85	16.11	16.37	16.63	i 6.89	17.15	17.41	17.67	17.93	18.19	18.45	18.71	18.97	19.23	19.49	19.75	20.01	20.27	20.54	20.80
	27.5	•	•	10.41	10.03	61.11	11.45	17.71	11.97	12.23	12.49	12.75	13.01	13.27	13.53	13.79	14.06	14.32	14.58	14.84	15.10	15.36	15.62	15.88	16.14	16.40	16.66	16.92	17.19	17.45	17.71	17.97	18.23	18.49	18.75	19.01	19.27	19.53	19.80	20.06	20.32	20.58	20.84
	23.0	•	:	10.43	10.05	11.21	11.47	11.73	12.00	12.26	12.52	12.78	13.04	13.30	13.56	13.82	14.08	14.35	14.61	14.87	15.13	15.39	15.65	16.51	16.18	16.44	16.70	16.96	17.62	17.48	17.74	18.00	18.27	18.53	18.79	19.05	19.31	19.57	19.84	20.10	20.36	20.62	20.88
1	22.5			10.45	10.07	11.24	11.50	11.76	12.02	12.28	12.54	12.81	13.02	13.33	13.59	13.85	14.11	14.38	14.64	14.90	15.16	15.42	15.69	15.95	16.21	16.47	16.73	17.00	17.26	17.52	17.78	18,04	18.31	18.57	18.83	60.61	19.35	19.62	19.88	20.14	20.40	20.66	20.93
•	22.0			10.47	11.00	11.26	11.52	11.78	12.05	12.31	12.57	12.83	13.09	13.36	13.62	13.88	14.14	14.41	14.67	14.93	15.19	15.46	15.72	15.98	16.24	16.51	16.77	17.03	17.29	17.56	17.82	18:08	18.35	18.61	18.87	19.13	19.40	19.66	19.92	20.18	20.45	20.71	20.97
mtinued.)	21.5		1	10.49	11.02	11.28	11.55	11.81	12.07	12.33	12.60	12.86	13.12	13.39	13.65	13.91	14.17	14.44	14.70	14.96	15.23	15.49	15.75	16.01	16.28	16.54	16.80	17.07	17.33	17.59	17.86	18.12	18.38	18.65	18,91	19.17	19.44	19.70	19.96	20.22	20.49	20.75	
	Brix	Sugar Sugar	200	10.52	11.04	11.31	11.57	11.83	12.09	12.36	12.62	12.88	13.15	13.41	13.68	13.94	14.20	14.47	14.73	14.99	15.26	15.52	15.78	16.05	16.31	16.57	16.84	17.10	17.37	17.63	17.89	18.16	18.42	18.68	18.95	19.21	19.48	19.74	20.00	20.27			
TABLE	20.5	P.	3	10.54	11.02	11.33	11.59	11.86	12.12	12.39	12.65	12.91	13.18	13.44	13.70	13.97	14.23	14.50	14.76	15.02	15.29	15.55	15.82	16.08	16.35	16.61	16.87	17.14	17.40	17.67	17.93	18.19	18.46	18.72	18.99	19.25	19.52	19.78					
	20.0		,	10.50	11.00	11.35	11.62	11.88	12.15	12.41	12.67	12.94	13.20	13.47	13.73	14.00	14.26	14.53	14.79	15.06	15.32	15.58	15-85	16.11	16.38	16.64	16.91	17.17	17.44	17.70	17.97	18.23	18.50	18.76	19.03								
	10.5	7	•	10.58	11.11	11.38	11.64	16.11	12.17	12.44	12.70	12.97	13.23	13.50	13.76	14.03	14.29	14.56	14.82	15.09	15.35	15.62	15.88	16.15	16.41	16.68	16.94	17.21	17.47	17.74	18.00	18.27	18.53										
	10.0	•	,	0.01	11.13	11.40	11.66	11.93	12.20	12.46	12.73	12.99	13.26	13.52	13.79	14.05	14.32	14.59	14.85	15.12	15.38	15.65	15.92	16.18	16.45	16.71	16.98	17.24	17.51	17.78	18.04	18.31						•		•			
	8	;	,	10.62	11.16	11.42	11.69	11.96	12.22	12.49	12.75	13.02	13.29	13.55	13.82	14.08	14.35	14.62	14.88	15.15	15.42	15.68	15.95	16.21	16.48	16.75	17.01	17.28	17.55	17.81								٠,		٠			
	18.0		,	10.04	11.18	11.45	11.71	11.98	12.25	12.51	12.78	13.05	13.31	13.58	13.85	14.11	14.38	14.65	14.91	15.18	15.45	15.71	15.98	16.25	16.52	16.78	17.03	17.32				:											

Prix 7.88 Purity 1.88 Purity 1 0.01.00 0.0 Z. 7. 42244447277666666666666666711144486644448

- TABLE IV.—(Continued.)
- 786 0 1 2 2 4 4 7 7 8 9 0 0 1 2 4 7 8 7 9 0 1 2 2 2 2 2 2 3 4 4 7 8 9 0 1 2 2 4 4 7 8 9 0 1 2 2 4 4 7 8 9 0 1 2 3 4 4 7 8 9 0 1 2 3 4 7 8 9 0 1 2 3 4 7 8 9

9.7 

		11.0		48.3	49.I	50.0	50.9	51.8	52.7	53.6	54.5	55.5	56.4	57.3	58.3	59.1	90.0	600	8.19	62.7	63.6	64.5	65.5	66.4	67.3	68.3	1.69	70.0	70.9	71.8	72.7	73.0	74.5				70.2		80.0	80.0	8.1.8	25.7	200	2.5
		10.9		48.6	49.5	50.5	51.4	52.3	53.2	54.1	55.0	26.0	56.9	57.8	58.7	29.6	9.09	61.5	62.4	63.3	64.2	65.1	1.99	67.0	67.9	68.8	69.7	20.6	71.6	72.5	73.4	74.3	75.2		1,7,1	0.0	0.0	o.	80.7	81.0 0.18	82.0	93.5	4.4	03:0
•	Brix	10.8	Purity	49.1	50.0	50.0	51.9	52.8	53.7	54.6	55.6	56.5	57.4	58.3	59.3	60.2	61.1	62.0	63.0	63.9	8.49	65.7	2.99	9.29	68.5	69.4	70.4	71.3	72.2	73.1	74.1	75.0	75.9	, ,	0 10	/0/	29.0	0.00	81.5	82.4	63.3	04.3 6.4.3	200	:
		10.7		49.5	50.5	51.4	52.3	53.3	54.2	55.1	26.1	57.0	57.9	58.9	8.65	60.7	61.7	62.6	63.6	64.5	65.4	66.4	67.3	68.2	69.2	1.07	71.0	72.0	72.9	73.8	74.8	75.7	70.0	20.0	70.5	4.6	80.4	61.3	85.2	83.2	84.1	85.0 85.0	80.0	*
		10.6		50.0	50.9	51.9	52.8	53.8	54.7	55.7	26.6	57.5	58.5	59.4	60.4	61.3	62.3	63.2	64.2	65.1	0.99	67.0	6.79	689	8.69	70.8	7.1.7	72.6	73.6	74.5	75.5	70.4	77.4	70.3	6,0	7.00	81.1	02.1	83.0	84.0	× 6	0 0 0 0 0 0 0 0 0	87.7	.,
ed.)			Sug.	5.3	5.4	5.5	Ş.	5.7	œ.	5.9	0.9	6.1	6.2	6.3	6.4	6.5	9.9	6.7	8.	6.9	2.0	7.	7.5	7.3	4.7	7.5	2.6	7:7	7.8	2.0	ò	×.	000		4 .	o o	o .	, 0	œ œ	œ œ	0.6	9.	, c	?
—(Continued.)			P.R.	19.3	19.7	20.I	20.4	20.8	21.2	21.5	21.9	22.3	22.6	23.0	23.4	23.7	24.1	24.5	24.8	25.2	25.5	25.9	26.3	26.6	27.0	27.4	27.7	28.1	28.5	28.8	20.5	20.0	29.0	30.3	30.7	31.0	31.4	31.8	32.1	32.5	32.8	33.2	33.0	٧٠٠٥
rable IV.	_	10.5	,	47.6	9.8	49.5	50.5	51.4	52.4	53.3	54.3	55.2	56.2	52.1	58.1	20.0	0.00	0.1.0	6I.9	62.9	63.8	8.4.8	65.7	66.7	9.29	9.89	69.5	70.5	71.4	72.4	73.3	74.3	75.2	70.2	77.1	1.0/	20.0	0.00	0.10	6.16	0.0	000	0.4.0	
T		10.4		48.1	49.0	50.0	51.0	51.9	52.9	53.8	54.8	55.8	26.7	57.7	58.7	29.6	90.0	61.5	62.5	63.5	64.4	65.4	66.3	67.3	68.3	69.3	70.2	71.2	72.1	73.1	74.0	75.0	0.9	70.9	77.0	0.0	26.8	20.0	81.7	82.7	83.7	04.0	0.50	00.3
	Brix	10.3	Purity	48.5	49.5	50.5	51.5	52.4	53.4	54.4	55.3	56.3	57.3	.58.3	59.2	60.2	61.2	62.1	63.1	64.1	65.0	0.99	0.70	08.0	6.89	6.69	70.9	71.8	72.8	73.8	74.8	75.7	70.7	///	70.0	, c	90.0	01.0	82.5	83.5	8.4.5 S.4.5	45.4	4. 4	<b>t</b> :/0
		10.2		49.0	50.0	51.0	52.0	52.9	53.9	54.9	55.9	56.9	57.8	58.8	8.65	8.00 ,	61.8	62.7	63.7	64.7	65.7	2.99	9.29	9.89	9.69	20.6	9.1.6	72.5	73.5	74.5	75.5	70.5	4.67	70.4	4.60	4.00	81.4	62.3	83.3	84.3	85.3 85.3	60°	20.00	
		10.1		49.5	50.5	51.5	52.5	53.5	54.5	55.4	56.4	57.4	58.4	59.4	60.4	61.4	62.4	63.4	64.4	65.3	66.3	67.3	68.3	69.3	70.3	71.3	72.3	73.3	74.3	75.2	76.2	77.2	78.2	2.6	00.7	7.10	82.2	63.2	84.2	25.1	20.1	87.1	200.1	•
			Sug.	5.0	5.1	5.2	5.3	5.4	5.5	2.6	5.7	χ. 80.	5.9	0.0	9.1	6.2		6.4		9.9	6.7	<b>8</b> .	6.9	7.0	7.1	7.2	7.3	7.4	7.5	2.6	7.7	7.0	6.0	9		, 0	, ,	4:	x,	9.0	× o	xo o	ò 6	, ,
			P.R.	18.2	18.6	18.9	19.3	19.7	20.0	20.4	20.7	21.1	21.5	21.8	22.2	22.0	22.9	23.3	23.7	24.0	24.4	24.8	25.1	25.5	25.8	26.2	26.6	56.9	27.3	27.7	280	40.6	28.8	29.1	20.0	29.0	30.2	30.0	30.9	31.3	31.7	32.0	32.4	32.0

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33.53

	1.225	r e	12.0		48.3	49.3	20.0	20.8	51.7	52.5	53.3	54.2	55.0	55.0	50.7	57.5	58.3	20.5	00.0	80.0	61.7	62.5	63.3	64.2	05.0	0.5.0 0.5.0	200.7	07.5	9		200	71.7	72.5	73.3	74.2	74.0	80.5	16.7	77.5	78.3	79.3	•
	1.222	40.1	11.9		48.7	49.6	50.4	51.3	52.1	52°0	53.8	54.0	55.5	50.3	25.1	28.0	58.8	59.7	60.5 0	61.3	62.3	63.0	63.9	64.7	65.5	400	97.2	1.00	8 4 9 1	, (c	7 1.	72.3	72.1	73.0	8 4 7	75.6	76.5	77.3	78.2	20.0	79.8	<u>.</u>
	1.220	4/·/ Brix	8.11	Purity	49.2	50.0	50.8	51.7	52.5	53.4	54.2	55.1	55.0	50.0	0.79	50.5	59.3	60.2	61.0	6.19	62.7	63.6	64.4	65.3	1.00	67.0	8,2	0.00		? .		72.0	72.7	9.4.6	7.5.	, ,	77.1	20.	78.8	79.7	80.5	•
	1.218	47.3	11.7		49.6	50.4	51.3	52.1	53.0	23.8	54.7	25.0	50.4	57.3	20.	26.0	20.0	00.7	61.5	62.4	63.2	64. I	65.0	65.8	66.7	67.5	4.80	2.60		, <u>;</u>	22.6	72.5	7.7	75.2	1.92	26.0	200	200	70.5	80.3	81.2	
	-	0.0	9.11		\$0.0	80.9	21.7	52.6	53.4	54.3	55.2	20.0	50.0	57.0	20.0	59.5	8,	61.2	62.I	62.9	63.8	64.7	65.5	4.99	67.2		000	0.00	· ·	2,5	† °	17.	2 2 2	78.0	1.0	9.44	78.0		80.2	81.0	81.0	<b>b-</b>
•	Sp. gr. of und. syp.	unu. syp.		Sug.	5.8	5.9	••	6.1	6.2		4.0	o'	0	0.7	0.0	6.0	2.0	7.1	7.3	7.3	7.4	7.5	2.6	7:7	2.00	6.	o :			? a	t u	o c	2 00	·α	0		À		, 6	. 4	9.0	<b>&gt;</b>
TABLE IV.—(Continued.	Sp. gr.	DITA OI	•	P.R.	21.2	21.6	22.0	22.3	22.7	23. I	23.4	23.8	24.2	24.5	24.9	25.2	25.0	20.0	26.4	26.7	27.1	27.5	27.8	28.7	28.6	28.9	29.3	29.7	30.0	400		21.6		2.5	22.6		9.50		24.1	34.4	8772	,
BLR IV.			11.5		8. 24	48.7	49.0	50.4	51.3	52.2	53.0	,	9.1	7.7.	2	4.70	50.3	59.1	0.00	60.0	61.7	62.0	63.5	64.3	65.2	. P	0,0	8.2	, oo ,	2,0	1 2	20.00	4,5	73.0	2 4	1 2	7.0		18.7	70.1	0.00	i
Ţ		٠	11.4		48.2	49.1	20.0	50.9	51.8	52.0	53.5	4.4	5.5	100	2.7	, , , ,	20.0	29.0	00.	or.4	62.3	63.2	0.4.0	64.9	65.8	66.7	67.5	4.80	60 60 60 60 60 60 60 60 60 60 60 60 60		:::	70.	72.7	7.5.	7 26	1,4	2 6		280	70.0	80.7	
		Brix	11.3	Purity	48.7	49.5	50.4	51.3	52.2	53.1	54.0	 	25.0	0.00	.,	50.4	59.3	200.5	1.10	61.9	62.8	63.7	64.6	65.5	66.4	67.3	08.1	0.00	9,0	0 1	,,,	72.6		2.4.	1.04	1	2,7	, a	20.0	, o	71	-
			11.2		49.1	50.0	50.9	51.8	52.7	53.0	54.5	55.4	50.2	57.1	9	50.0	29.9	, 00.7	61.6	62.5	63.4	64.3	65.2	1.99	67.0	62.0	2.7	0.60	70.5	4:4	?;;			1,0	, c	1 2	12/2	1	0.4	81.2	82.1	i.
			11.1		49.5	50.5	51.4	52.3	53.2	54.1	55.0	55.0	20.0	57.7	20.0	59.5	4.00	o1.3	62.2	63.1	04.0	64.9	65.8	2.99	9.29	68.5	4.60	70.3	71.2	72.1	, t	, , ,	1 1	, v	u 0	200	4 0 0	? ¢	1.10	82.0	200	<b>k</b>
				Sug.	5.5	2.6	5.7	5. 80	0.	٠ •		7 °	۳ • د	4.0	o.	0.0	, ,	×.	6.9	2.0	7.1	7.2	7.3	7.4	7.5	2.6	7.7	2.8	6.0			e e	? <b>.</b>	t u	n v	) a	, a	o c	ý	) i		<b>.</b>
				P.R.	20.1	20.5	20.9	21.2	21.6	21.9	22.3	22.7	23.0	4.5.	23.0	24.1	24.5	24.9	25.3	25.6	26.0	26.3	26.7	27.1	27.4	27.8	28.1	28.5	20.9	29.2	9,0	200	2 6	30.7	31.1		31.0	4	22.0	V	3.5	2

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885.5. 885.5. 885.5. 885.6. 887.7. 887.7. 887.6.

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.248 52.7 .246 52.3 12.9 1.243 1.102 1.102 1.102 1.102 1.102 1.102 1.102 1.103 1. 1.241 51.5 1.239 51.1 12.6 Sp. gr. of und. syp. Brix of und. syp. syp. FABLE IV.—(Continued.) 1.236 50.6 2.5 1.234 50.2 12.4 Furia 18.38 18 49.4 12.2 1.227 48.9 12.1 Sp. gr. of und. syp. Brix of und. syp. 

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	1.269	56.5	13.9		48.9	49.6	50.4	51.1	51.8	52.5	53.2	54.0	54.7	55.4	56.1	20.8	57.6	58.3	59.0	59.7	<b>60.4</b>	61.2	6.19	62.6	63.3	04.0	64.7	65.5	66.2	6.99	9.29	8.3	1.69	8.69	70.5	71.2	71.9	72.7	73.4	74.1	74.8
	1.267	56.0 Brix	13.8	Purity	49.3	20.0	20.7	51.4	52.2	52.9	53.6	54.3	55.1	55.00	56.5	57.2	58.0	58.7	59.4		60.0	9.19	62.3	63.0	63.8	64.5	65.2	62.0	66.7	67.4	68.1	8.8	9.69	70.3	71.0	71.7	72.5	73.2	73.9	74.6	75.4
	1.264	\$5.6	13.7		49.6	50.4	51.1	51.8	52.6	53.3	54.0	54.7	55.5	50.2	56.9	57.7	58.4	59.1	59.0	9.09	61.3	62.0	62.8	63.5	64.2	65.0	65.7	66.4	67.2	6.79	9.89	69.3	70.1	70.8	71.5	72.3	73.0	73.7	74.5	75.2	75.9
	1.262	. 55.2	13.6		50.0	50.7	51,5	52.2	52.9	53.7	54.4	55.1	55.9	50.0	57.4	58.1	58.8	59.6	60.3	01.0	8.19	62.5	63.2	64.0	64.7	65.4	66.3	6.9	9.29	68.4	1.69	66.6	20.6	71.3	72.1	72.8	73.5	74.3	75.0	75.7	29.2
d.)	Sp. gr. of und. syp.	und. syp.		Sug.	8.9	6.9	7.0	7.1	7.5	7.3	7.4	7.5	7.6	7:7	7.8	7.9	<b>8</b> .0	<b>8</b> 0	8.5	8.3	<b>8</b>	8.5	9.8	8.7	8. 8.	8.9	0.6	1.6	9.5	9.3	4.6	o S	9.6	2.6	ن ه	6.6	10.0	10.1	10.2	10.3	10.4
IV.—(Continued.)	Sp. gr. of	Brix of		P.R.	25.1	25.5	25.9	26.3	26.7	27.1	27.4	27.7	28.1	28.5	28.8	1.62	29.5	29.0	30.3	30.7	31.0	31.4	31.8	32.2	32.6	32.9	33.2	33.6	34.0	34.4	34.7	35.1	35.5	35.9	36.3	36.6	36.9	37.3	37.7	38.0	38.4
TABLE IV	1.260	54.8	13.5		48.9	49.6	50.4	51.1	51.9	52.6	53.3	54.1	8.4.8	55.0	56.3	57.0	57.8	58.5	59.3	0.0	60.7	61.5	62.2	63.0	63.7	4.4	65.2	62.0	66.7	67.4	1.89	68.0	9.69	70.4	71.1	71.9	72.6	73.3	74.1	74.8	75.6
TA	1.257	54.4	13.4		49.3	50.0	20.7	51.5	52.2	53.0	53.7	54.5	55.2	20.0	26.7	57.5	58.2	59.0	59.7	60.4	61.2	61.9	62.7	63.4	64.2	64.9	65.7	60.4	67.2	67.9	68.7	69.4 4.6	70.1	70.9	9.1.6	72.4	73.1	73.9	74.6	75.4	76.1
	1.255	53.9 Brix	13.3	Purity	49.6	50.4	51.1	51.9	52.6	53.4	54.1	54.9	55.6	50.4	57.1	57.9	58.6	59.4	60.2	6.00	61.7	62.4	63.2	63.9	64.7	65.4	66.2	6.9	67.7	68.4	69.2	6.69	70.7	71.4	72.2	72.9	73.7	74.4	75.2	75.9	76.7
	1.252	53.5	13.2		50.0	50.8	51.5	52.3	53.0	53.8	54.5	55.3	26.1	20.8	27.6	58.3	29.I	8.65	9.09	61.4	62.1	62.0	63.6	64.4	65.2	6.5	2.99	67.4	68.3	6.89	69.7	70.5	71.2	72.0	72.7	73.5	74.2	75.0	75.8	76.5	77.3
	1.250	53.1	13.1		50.4	51.1	51.9	52.7	53.4	54.2	55.0	55.7	50.5	57.3	58.0	58.8	59.5	60.3	61.1	8.19	62.6	63.4	64.1	64.9	65.6	66.4	67.2	62.9	68.7	69.5	70.2	71.0	71.8	72.5	73.3	74.0	74.8	75.6	76.3	77.1	77.9
	Sp. gr. of und. syp.	f und. syp.		Sug.	9.9	6.7	8.9	6.9	2.0	7.1	7.3	7.3	7.4	7.5	2.6	7:7	7.8	6.2	8.o	<b>8</b> .1	8.3	.3 .3	8.4	.S.	9.6	8.7	8. 8.	8. 6.	0.6	1.6	9.5	9.3	4.6	9.2	9.6	6.7	80.0	6.6	10.0	10.1	10.2
	Sp. gr.	Brix of und.		P.R.	24.3	24.7	25.I	25.5	25.8	26.1	26.5	26.9	27.3	27.0	28.0	28.4	28.8	29.5	29.5	29.9	30.3	30.7	31.0	31.3	31.7	32.0	32.4	32.8	33.2	33.6	34.0	34.3	34.6	35.0	35.4	35.8	36.1	36.5	36.8	37.2	37.6

75.0	75.7	76.4	77.1	77.9	78.6	79.3	80.0	80.7	81.4	82.1	82.9	83.6	84.3	85.0	85.7	86.4	87.1	87.9	88.0	89.3	90.0	90.7	91.4	92.1
75.5	76.3	77.0	77.7	78.4	79.1	29.9	80.6	81.3	82.0	82.7	83.5	84.2	84.9	85.6	86.3	87.1	87.8	88.5	89.2	89.9	9.06	91.4	92.1	92.8
76.1	76.8	77.5	78.3	29.0	79.7	80.4	81.2	8I.9	82.6	83.3	84.1	84.8	85.5	86.2	87.0	87.7	88.4	89.1	89.9	90.0	91.3	92.0	93.8	93.5
9.92	77.4	78.1	78.8	29.6	80.3	81.0	81.8	82.5	83.2	83.9	84.7	85.4	86.1	86.9	87.6	88 89.3	1.68	89.8	90.5	91.2	92.0	92.7	93.4	94.2
77.2	77.9	78.7	79.4	80.1	80.0	81.6	82.4	83.1	83.8	84.6	85.3	86.0	86.8	87.5	88.2	8 0.68	89.7	90.4	91.2	91.9	93.6	93.4	1.76	94.9
10.5	10.6	10.7	10.8	10.9	11.0	11.1	11.2	11.3	11.4	11.5	911	11.7	8.11	6.11	12.0	13.1	12.2	12.3	12.4	12.5	12.6	12.7	12.8	12.9
38.7	39.1	39.8	39.9	40.3	40.6	41.0	41.4	41.8	42.I	42.5	42.9	43.3	43.6	43.9	44.3	44.7	45.1	45.5	45.9	46.2	46.6	47.0	47.3	47.6
76.3	77.0	27.8	78.5	79.3	80.0	80.7	81.5	82.2	83.0	83.7	84.4	85.2	85.9	86.7	87.4	88.1	88.0	89.6	90.4	91.1	91.9	95.6	93.3	1.46
76.9	27.6	78.4	. 1.62	79.9	80.6	81.3	82.1	82.8	83.6	84.3	85.1	82.8	9.98	87.3	88.1	88.8	9.68	90.3	91.0	91.8	92.5	93.3	94.0	94.8
77.4	78.3	78.9	79.7	80.5	81.2	82.0	82.7	83.5	84.2	85.0	85.7	86.5	87.2	88.0	88.7	89.5	90.2	91.0	91.7	92.5	93.2	94.0	94.7	95.5
78.0	78.8	79.5	80.3	81.1	81.8	82.6	83.3	84.1	84.8	85.6	86.4	87.1	87.9	88.6	89.4	5005	6.06	7.16	92.4	93.2	93.9	94.7	95.5	5.96
78.6	79.4	80.2	80.9	81.7	82.4	83.2	84.0	84.7	85.5	86.3	87.0	87.8	88.6	89.3	1.06	8.06	9.16	92.4	93.1	93.9	94.7	95.4	96.2	6.96
10.3	10.4	10.5	10.6	10.7	10.8	10.9	0.11	11.1	11.2	11.3	11.4	11.5	9.11	11.7	8.11	6.11	12.0	12.1	12.2	12.3	12.4	12.5	12.6	12.7
37.9	38.3	38.7	39.1	39.5	39.8	40.2	40.5	40.9	41.3	41.7	42.0	43.4	42.8	43.2	43.6	43.9	44.2	44.6	45.0	45.3	45.7	46.1	46.5	46.8 46.8

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IV.—(C
TABLE

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	1.298	61.3	15.0	48.7	49	50.0	50.	51.	52.0	52.	25.	24.0	54.7	55.	200	9	200	ģ		à c				200		3.2	Š	,	ė v	3	3	200	8	Š	6	20.	70.	71.	72.	72.
•	1.295	60.8	14.9	49.0	49.7	50.3	51.0	21.7	52.3	53.0	53.7	54.4	55.0	25.7	50.4	57.0	28.7	50.4		7			7.7	4.20	1.0	2.50	4.4		0.20	4.00		82.8	08.5	1.66	8.60	70.5	71.1	71.8	72.5	73.2
	1.292	60.4 Brix	14.8 Purity	49.3	50.0	50.7	51.4	52.0	52.7	53.4	54.1	54.7	4.55	20.1	0.0	57.4	20.1	20.0	5.65	1.00	2.50		7.7	02.0	03.5	4.5	, 1 v	6,50	200	6.00	02.0	2.5	08.0	9.69	70.3	70.9	21.6	72.3	73.0	73.6
	1.290	0.09	14.7	40.7	50.3	51.0	51.7	52.4	53.1	53.7	54.4	55.	55.0	50.5	57.1	57.0	50.5	59.2	59.9	500.5	7.10	y 2	02.0	03.3	03.0	0.4.0	5.5	9.0	7.00	07.3	0.00	68.7	4.69	70.1	70.7	71.4	72.1	72.8	73.5	74.1
	1.288	. 9.65	14.6	50.0	50.7	51.4	52.1	52.7	53.4	54.1	54.8	55.55	50.2	50.0	57.5	20.7	20.0	59.0	00.3	01.0	0.10	5.5	03.0	03.7	4.40	25.1	0.5.0	4.00		0.20	9.5	69.2	6.69	70.5	71.2	71.9	72.6	73.3	74.0	74.7
ea.)	f und. syp.	und. syp.	Sug.	7.3	4.	7.5	9.2	7:7	7.8	6.	0	 	0.5		4.	io o	0.0	, o	0.0	0.0	9.	- -	9.5	9.3	4.6		9.0	, o	0	6.6	10.0	10.1	10.2	10.3	10.4	10.5	9.01	10.7	8.01	10.9
-(Communication)	Sp. gr. of und. 8	Brix of	ъ. В.	27.1	27.4	27.8	28.7	28.6	29.0	29.3	29.7	30.1	30.5	30.8	31.1	31.5	31.9	32.3	32.7	33.0	4.5.	33.0	34.1	34.5	34.8	35.2	35.0	30.0	30.4	30.7	37.1	37.5	37.9	38.3	38.6	38.9	30.3	39.7	40.1	40.4
VBLE 1V.	1.285	59.1	14.5	48.3	60.64	49.7	50.3	51.0	51.7	52.4	53.1	53.8	54.5	55.2	55.9	20.0	57.2	52.0	50.0	59.3	0.00	200	4.10	02.1	02.8	63.4	04.1	8.50	5.50	60.3	6.09	9.29	68.3	0.09	60.7	70.3	71.0	71.7	72.4	73.1
7.	1.282	58.7	14.4	48.6	49.3	20.0	50.7	51.4	52.1	52.8	53.5	54.2	54.9	55.6	50.2	56.9	57.6	58.3	20.0	29.7	4.00	1.10	8.19	62.5	63.2	63.9	64.6	05.3	00.0	66.7	67.4	1.89	68.7	69.4	70.1	70.8	71.5	72.2	72.0	73.6
	1.280	58.3 Brix	14.3 Purity	40.0	49.7	50.3	51.0	51.7	52.4	53.1	53.8	54.5	. 55.2	55.0	20.0	57.3	58.0	58.7	59.4	.00 ,	8.00	61.5	62.2	62.9	63.6	64.3	62.0	65.7	66.4	67.1	67.8	68.5	69.2	60.0	20.6	71.3	72.0	72.7	73.4	74.1
		<b>6</b> 0	~	,	, 0	7	. 4	-	œ.	S		o,	ø.	ě	o.	٠.	ż	Ġ	٠. و	9.0		5.0	2.7	3.4		×.	iv.	6.2	6.9	2.6		.0	7.0	4.	3	<b>8</b> :		2 0	0.0	74.6
	1.2	57.8	14.2	40	0	0.00	51	52	52	23	Ŋ,	54	55	20	22	55	Š	8	χ,	ŏ١	ō	Ö	•	•	ď.	•	Ö	۰ ق	Ō	•	3	Ğ	Ö	7				4		
	1.27 5 1.2		14.1 14.2																																					75.2
	syp. 1.275	57.4	14.1		50.4	51.1	21.8	52.5	53.2	53.9	54.6	55.3	26.0	26.7	57.4	5.00	58.9	29.6	60.3	0.19	61.7	62.4	63.1	63.8	64.5	65.2	0.0	66.7	67.4		68.8	69.5	70.2	20.0	21.6	72.3	7.7	2 2	74.5	

7.77 7.78 7.88 7.89 

	1.323 65.6	,	10.0		48.7	49.4	20.0	20.0	51.2	51.9	52.5	53·I	53.7	54.4	55.0	55.6	56.3	56.9	57.5	58.1	58.7	59.4	0.0	9.09	61.2	61.9	62.5	63.1	03.7	04:4	65.0	65.6	66.2	6.9	67.5	68.1	68.7	4.69	70.0	70.0
	1.321 65.2		15.9		1.64	49.7	50.3	50.9	21.6	52.2	52.8	53.5	54.1	54.7	55.3	26.0	26.6	57.2	57.9	58.5	1.65	26.7	60.4	61.0	9.19	62.3	62.0	63.5	64.2	8.4.8	65.4	0.99	66.7	67.3	62.9	9.89	69.2	8.69	70.4	71.1
	1.319 64.8	Brix	15.8	Purity	49.4	50.0	20.6	51.3	51.9	52.5	53.2	53.8	54.4	55. I	55.7	56.3	57.0	57.6	58.2	58.9	59.5	60.1	8.09	<b>61.4</b>	62.0	62.7	63.3	63.9	04.0	65.2	65.8	66.5	67.1	67.7	<b>4</b> .89	0.69	9. <b>6</b> 9	70.3	70.9	71.5
	1.316 64.4		15.7		49.7	50.3	51.0	91.6	52.2	52.9	53.5	54.1	54.8	55.4	56.1	26.7	57.3	58.0	58.6	59.3	59.9	<b>5</b> .09	61.1	61.8	62.4	63.1	63.7	64.3	65.0	02.0	00.2	6.99	67.5	68.2	8.8	69.4	70.1	70.7	71.3	72.0
	1.313 63.9		15.6		50.0	50.6	51.3	51.9	52.6	53.2	53.8	54-5	55. I	55.8	56.4	57.1	57.7	58.3	59.0	29.6	60.3	6.09	61.5	62.2	62.8	63.5	64.1	64.7	65.4	00.0	60.7	67.3	62.0	9.89	69.2	6.69	70.5	71.2	71.8	72.4
eed.)	of und. syp.			Sug.	7.8	7.9	8.0	 	8.2	.3	<b>8</b> 0	so o	٠ •	8.7	90 90	8. 0.8	0.6	1.6	9.5	9.3	9:4 4:0	9.5	9.6	2.6	œ.	6.6	10.0	10.1	10.2	10.3	10.4	10.5	9.01	10.7	10.8	10.9	0.11	11.1	11.2	11.3
$\overline{}$	Sp. gr. of und. Brix of und.			P.R.	29.0	29.4	29.8	30.2	30.6	30.9	31.3	31.6	32.0	32.4	32.8	33.1	33.5	33.9	34.2	34.6	35.0	35.4	35.8	36.1	36.5	30.9	37.2	37.6	38.0	38.3	38.7	39.1	39.2	39.9	40.3	40.7	41.0	41.4	41.8	42.1
TABLE IV	1.310		15.5		49.0	49.7	50.3	51.0	51.6	52.3	52.9	53.5	54.2	54.8	55.5	26.1	26.8	57.4	58.1	58.7	59.4	0.09	9.09	61.3	6.19	9.29	63.2	63.9	64.5	65.2	65.8	99.	67.1	67.7	68.4	0.69	69.7	70.3	71.0	71.6
TA	1.308 63.0		15.4		49.4	50.0	50.6	51.3	51.9	52.6	53.2	53.9	54.5	55.2	55.8	56.5	57.1	57.8	58.4	59.1	59.7	60.4	61.0	61.7	62.3	63.0	63.6	64.3	64.9	65.6	66.2	6.99	67.5	68.2	68.8	69.5	70.1	70.8	71.4	72.1
	1.305 62.6	Brix	15.3	Purity	49.7	50.3	51.0	51.6	52.3	52.9	53.6	54.2	54.9	55.6	56.2	56.9	57.5	58.2	58.8	59.5	1.09	8.09	61.4	62.1	62.7	63.4	64.1	64.7	65.4	0.99	2.99	67.3	68.0	9.89	69.3	6.69	9.0%	71.2	71.9	72.5
	1.303		15.2		50.0	50.7	51.3	52.0	52.6	53.3	53.9	54.6	55.3	55.9	26.6	57.2	57.9	58.6	59.2	59.0	60.5	61.2	61.8	62.5	63.2	63.8	64.5	65.1	65.8	66.4	67.1	67.8	68.4	1.69	69.7	70.4	71.0	71.7	72.4	73.0
	1.301		15.1		50.3	51.0	51.7	5.5.3	53.0	53.6	54.3	55.0	55.6	56.3	57.0	27.6	58.3	58.0	59.6	60.3	6.09	9.19	62.3	62.9	63.6	64.2	64.9	65.6	66.2	6.99	67.5	68.2	68.9	60.5	70.2	20.9	71.5	72.2	72.8	73.5
	of und. syp.			Sug.	2.6	7.7	. 00	2.0	. 00	8.1	8.8	80	œ 4	. so	9.6	8.7	œ œ	8.0	0.0		6.6	9.3	4.6	55.	96	7.6	8.0	9	10,0	10.1	10.2	10.3	10.4	10.5	10.6	10.7	10.8	10.9	11.0	11.1
	Sp. gr. of Brix of			P.R.	28.2	28.6	20.0	20.3	20.7	30.1	30.5	30.0	31.3	31.6	32.0	32.4	32.8	11.1	33.4	33.8	34.2	34.6	34.0	35.3	35.7	36.1	36.5	36.8	17.1	37.5	37.0	38.3	38.6	30.0	30.4	30.00	40.2	40.5	0.0	41.3

71.2	71.9	72.5	73.1	73.7	74.4	75.0	75.6	76.2	76.9	77.5	78.1	78.7	79.4	80.0	80.6	81.2	81.9	82.5	83.1	83.7	84.4	85.0	85.6	86.2	86.9	87.5	88.1	88.7	89.4	90.0	90.0	91.2	91.9	92.5
71.7	72.3	73.0	73.6	74.3	74.8	75.5	76.1	26.7	77.4	78.0	78.6	79.3	79.9	80.5	81.1	81.8	82.4	83.0	83.7	84.3	84.0	85.5	86.2	86.8	87.4	88.1	88.7	89.3	89.9	90.6	91.2	8.16	92.5	93.1
72.3	72.8	73.4	74.1	74.7	75.3	75.9	9.92	77.3	77.8	78.5	79.1	79.7	80.4	81.0	81.6	82.3	82.9	83.5	84.2	84.8	85.4	86.1	86.7	87.3	88.0	98.6	89.2	89.9	90.5	91.1	8.16	92.4	93.0	93.7
72.6	73.2	73.9	24.5	75.2	75.8	76.4	77.1	77.1	78.3	29.0	29.6	80.3	80.9	81.5	82.2	82.8	83.4	84.1	84.7	85.4	86.0	9.98	87.3	6.78	88.5	89.2	89.8	90.4	1.16	61.7	92.4	93.0	93.6	94.3
73.1	73.7	74.4	75.0	75.6	76.3	6.94	22.6	78.2	78.8	79.5	80.1	80.8	81.4	82.1	82.7	83.3	84.0	84.6	85.3	85.9	86.5	87.2	87.8	88.5	1.68	89.7	4.06	0.16	61.7	92.3	92.9	93.6	94.2	94.9
11.4	11.5	9.11	11.7	8.11	6.11	12.0	12.1	12.3	12.3	12.4	12.5	12.6	12.7	12.8	12.9	13.0	13.1	13.2	13.3	13.4	13.5	13.6	13.7	13.8	13.9	14.0	14.1	14.2	14.3	14.4	14.5	14.6	14.7	14.8
42.5	42.8	43.2	43.6	44.0	44.3	44.7	45.1	45.5	45.9	46.2	46.5	46.9	47.3	47.6	48.0	48.4	8.8	40.2	40.6	49.0	50.3	50.7	51.1	51.4	51.8	52.1	52.5	52.9	53.3	53.6	54.0	54.4	54.8	55.1
72.3	72.9	73.5	74.3	74.8	75.5	76.1	26.8	77.4	78.1	78.7	79.3	80.0	80.6	81.3	81.0	82.6	83.2	83.9	84.5	85.2	85.8	86.5	87.1	87.7	88.4	89.0	89.7	90.3	0.16	91.6	92.3	92.9	93.5	94.2
72.7	73.4	74.0	74.7	75.3	26.0	26.6	77.3	77.9	78.6	79.3	662	80.5	81.2	81.8	82.5	83.1	83.8	84.4	85.1	85.7	86.4	87.0	87.7	88.3	89.0	9.68	90.3	6.06	91.6	92.2	92.9	93.8	94.2	94.8
73.2	73.9	74.5	75.2	75.8	76.5	77.1	77.8	78.4	79.1	79.7	80.4	81.0	81.7	82.3	83.0	83.7	84.3	85.0	85.6	86.3	86.9	87.6	88.2	88.0	89.5	90.2	8.06	91.5	92.3	92.8	93.5	94.1	94.8	95.4
73.7	74.3	75.0	75.7	76.3	77.0	27.6	78.3	78.9	29.6	80.3	80.9	91.8	82.2	82.9	83.6	84.2	84.9	85.5	86.2	86.8	87.5	88.2	88.8	89.5	1.06	8.06	91.4	92.1	92.8	93.4	94.1	94.7	95.4	1.96
74.2	74.8	75.5	76.2	26.8	77.5	78.1	78.8	79.5	80.1	80.8	81.5	82.1	82.8	83.4	84.1	84.8	85.4	86.1	86.8	87.4	88.1	88.7	89.4	1.06	2006	91.4	92.1	92.7	93.4	94.0	94.7	95.4	96.0	96.7
11.2	11.3	11.4	11.5	9.11	11.7	8.11	6.11	12.0	12.1	12.2	12.3	12.4	12.5	12.6	12.7	12.8	12.9	13.0	13.1	13.2	13.3	13.4	13.5	13.6	13.7	13.8	13.9	14.0	14.1	14.3	14.3	14.4	14.5	14.6
11.7	12.0	13.4	12.7	†3.1	13.5	£3.8	4.2	9.41	12.0	<b>\$5.4</b>	15.7	9.0	4.9	8.9	17.2	17.5	6.21		18.7	1.64	49.4	8.6	50.2	9.0	51.0	51.3	91.6	2.0	2.4	8.5	3.2	3.5	3.9	54.2

	70.0	17.0	48.8	49.4	50.0	20.0	51.2	51.0	52.4	52.9	53.5	54.1	54.7	55.3	55.9	50.5	57.1	57.6	58.2	28.8	59.4	0.09	9.09	61.2	61.8	62.4	62.0	63.5	64.1	04.7	65.3	62.0	66.5	67.1	9.29	68.2	8.8	69.4	.0.07
	1.34 <b>8</b> 69.6	16.9	49.1	49.7	50.3	50.9	51.5	52. I	52.7	53.3	53.8	54.4	55.0	55.0	50.2	56.8	57.4	58.0	58.6	59.2	29.8	60.4	6.09	61.5	62.1	62.7	63.3	63.9	64.5	65.1	65.7	66.3	6.99	67.5	68.0	68.6	69.2	8.69	70.4
	1.346 69.2 Brix	16.8 Purity	49.4	50.0	50.6	51.2	51.8	52.4	53.0	53.0	54.2	54.8	55.4	20.0	50.5	57.1	57.7	58.3	58.9	59.5	1.09	60.7	61.3	6.19	62.5	63.1	63.7	64.3	64.9	65.5	66.1	66.7	67.3	62.9	68.5	0.69	9.69	70.3	20.8
	1.343 68.7	16.7	49.7	50.3	50.9	51.5	52.1	52.7	53.3	53.9	54.5	55.1	55.7	50.3	56.9	57.5	58.1	58.7	59.3	59.9	60.5	61.1	61.7	62.3	62.9	63.5	64.1	64.7	65.3	62.0	66.5	67.1	67.7	68.3	689	69.5	70.1	70.7	71.3
	1.340 68.3	16.6	50.0	50.6	51.2	51.8	52.4	53.0	53.0	54.2	54.8	55.4	56.0	26.6	57.2	57.8	58.4	59.0	29.6	60.2	8.09	61.4	62.0	62.7	63.3	63.9	64.5	65.1	65.7	66.3	6.99	67.5	1.89	68.7	69.3	60.0	70.5	71.1	7:12
ued.)	Sp. gr. of und. syp. Brix of und. syp.	S	 	& 4.	. SO	9.6	8.7	æ. •	6.9	0.6	9.1	9.5	. 9.3	9.4	9.8	9.6	7.6	8.6	6.6	10.0	10.1	10.2	10.3	10.4	10.5	9.01	10.7	10.8	10.9	0.11	11.1	11.2	11.3	11.4	11.5	9.11	11.7	11.8	11.9
Ĭ	Sp. gr. Brix o	9	31.0	31.4	31.8	32.2	32.6	33.0	33.3	33.6	34.0	34.4	34.8	35.1	35.5	35.9	36.3	36.7	37.0	37.4	37.8	38.2	38.6	38.0	30.3	39.7	40.0	40.4	40.8	41.1	41.5	41.9	42.3	42.7	43.0	43.4	43.8	44.2	44.5
TABLE IV.	1.337 67.8	. 16.5	48.5	40.1	49.7	50.3	50.9	51.5	52.1	52.7	53.3	53.9	54.5	55.2	55.8	26.4	57.0	57.6	28.5	8.8	50.4	0.00	909	61.2	819	62.4	63.0	63.6	64.2	64.8	65.5	1.99	66.7	67.3	6,79	68.5	69.1	69.7	70.3
4	1.334 67.3	16.4	48.8	40.4	20.0	50.6	51.2	51.8	52.4	53.0	53.7	54.3	54.9	55.5	1.95	56.7	47.3	2.7.5		50.1	80.8	4.00	61.0	919	62.5	62.8	63.4	64.0	64.6	65.2	62.0	66.5	67.1	67.7	68.3	68.0	69.5	70.1	70.7
	1.331	16.3	Furity 49.1	40.7	50.3	50.0	51.5	52.1	52.8	53.4	54.0	54.6	5.5	80.5	4.6	57.1	47.7		000	70.7	200	60.7	61.3	62.0	9.29	62.2	87.0	64.4	65.0	9.59	99	66.0	67.5		68.7	60.3	0.09	20.6	71.2
	1.32 <b>9</b> 66.5	16.2	707		50.6	51.2	51.0	2.5	53.1	53.7	54.3	9.45	92.6	20.5	8,9	7.7	28	9	20.0	0.00	200	1.0	61.7	, ,	?	9.69	64.2	64.8	65.4	99	66.7	67.3	62.0	280	60.1	809	70.4	71.0	71.6
	1.327 66.1	1 .91	40.7		20.0	51.6	22.2	87.8	53.4	54.0	54.7	55.3		46.5	1.7	2,78	× ×	100	200	200	9	, ,		2 2 2	7.79	1 0	949	65.2	8.8	299	67.1	67.7	68.3	89	909	20.0	200	71.4	72.0
	of und. syp. f und. syp.		Sug.	o c			, oc	œ	<b>.</b>	8.7	00	8.0	0.0				; c		, c	3 6	۰ ۵	9 6	× 5	9.0	1.01	7 01	5.01	9 01	9.01	10.7	10.01	10.01	011	: :	11.2	::	? :	7.11	11.6
	Sp. gr. of und. Brix of und.	1	가 6 자 6	,	30.6	20.0	21.2	21.7	12.1	32.5	22.0	33,2	22.6	24.0	***	, t	2 .	35.1	35.4	20.00	3,75	20.00	9.00	5./. 5.1	37.7	. o	. ec	0.00	30.0	0.04	40.4	70.7	) I V		α.	5.4	. · ·	75.0	43.3

•	,				;		۸٠.٠	?:					
4.4.6	92.9	93.5		4.0 0.0	15.7	50.7	5	9.50	24.5	95.1	, so	15.4	. v.
8.16	92.3	92.9	93.4	94.0	15.6	58.3	92.7	93.3	93.9	4.4	95.0	15.3	57.1
91.2	61.7	92.3	92.8	93.4	15.5	57.9	92.1	92.7	93.3	93.8	94.4	15.2	56.8
90.6	1.16	61.7	92.2	92.8	15.4	97.6	91.5	92.1	93.6	93.2	93.8	15.1	56.4
90.0	90.5	1.16	9.16	92.2	15.3	57.3	6.06	91.5	92.0	92.6	93.2	15.0	26.0
89.4	89.0	90.5	91.0	91.6	15.2	56.9	90.3	90.9	91.4	92.0	92.5	14.9	55.6
88. 88.	89.3	89.9	90.4	91.0	1.5.1	56.5	89.7	90.2	8.06	91.4	6.16	14.8	55.3
88.2	88.8	89.3	89.8	90.4	15.0	26.1	89.1	89.6	90.2	90.7	91.3	14.7	54.9
87.6	88.2	88.7	89.2	89.8	14.9	55.8	88.5	89.0	89.6	1.06	2.06	14.6	54.5
87.1	87.6	88.1	88.6	89.2	14.8	55.4	87.9	88.4	89.0	89.5	106	14.5	54.1
86.5	87.0	87.5	88.0	88.6	14.7	55.0	87.3	87.8	88.3	88.9	89.4	14.4	53.7
85.0	86.4	86.9	87.4	88.0	14.6	54.6	86.7	87.2	87.7	88.3	88.8	14.3	53.4
65.3	80 80	86.3	86.8	87.3	14.5	54.2	86.1	9.98	87.1	87.7	88.2	14.2	53.0
84.7	85.3	85.7	86.2	86.7	14.4	53.8	85.5	86.0	86.5	87.0	87.6	14.1	\$2.6
84.1	84.6	85.1	85.6	1,98	14.3	53.5	84.8	85.4	80	86.4	87.0	14.0	2.5
83.5	84.0	84.5	85.0	85.5	14.2	53.1	84.2	84.8	85.3	8.5	86.1	13.0	0.17
82.9	83.4	83.9	84.4	84.9	14.1	52.7	83.6	84.1	84.7	85.2	85.7	13.8	9:13
82.4	82.8	83.3	83.8	84.3	14.0	52.3	83.0	83.5	84.0	84.6	85.1	13.7	21.2
81.8	82,2	82.7	83.2	83.7	13.9	51.9	82.4	82.9	83.4	83.9	84.5	13.6	80.0
81.2	81.7	82.1	82.6	83.1	13.8	\$1.6	81.8	82.3	82.8	83.3	83.8	13.5	50.4
80.6	81.1	81.5	82.0	82.5	13.7	51.2	81.2	81.7	82.2	82.7	83.2	13.4	0.0
80.0	80.5	81.0	81.4	81.9	13.6	50.8	9.08	81.1	81.6	82.1	82.6	13.3	40.7
79.4	79.9	80.4	80.8	81.3	13.5	50.5	80.0	80.5	81.0	81.5	82.0	13.2	60.3
78.8	79.3	79.8	80.2	80.7	13.4	50.1	79.4	29.9	80.4	80.0	81.4	13.1	8.0
78.2	78.7	79.2	9.62	80.1	13.3	49.8	78.8	79.3	20.8	80.2	80.7	13.0	8
27.6	78.1	78.6	20.0	79.5	13.2	49.4	78.2	78.7	79.1	20.6	80.1	12.0	28,2
77.1	77.5	78.0	78.4	78.9	13.1	49.0	17.6	78.0	78.5	29.0	79.5	12.8	87.8
76.5	76.9	77.4	77.8	78.3	13.0	48.6	27.0	77.4	77.9	78.4	78.9	12.7	47.4
75.9	76.3	76.8	77.2	7.7.7	12.9	48.3	76.4	76.8	77.3	77.8	78.3	12.6	17.0
75.3	75.7	76.2	9.92	77:1	12.8	47.9	75.8	76.3	76.7	77.2	27.6	12.5	46.6
74.7	75.1	75.6	26.0	76.5	12.7	47.5	75.2	75.6	76.1	76.5	77.0	12.4	6.3
74.1	74.6	75.0	75.4	75.9	12.6	47.1	74.5	75.0	75.5	75.9	76.4	12.3	46.0
73.5	74.0	74.4	74.9	75.3	12.5	46.7	73.9	74.4	74.8	75.3	75.8	12.2	45.6
72.9	73.4	73.8	74.3	74.7	12.4	46.4	73.3	73.8	74.2	74.7	75.2	12.1	45.2
72.4	72.8	73.2	73.7	74.1	12.3	46.1	72.7	73.2	73.6	74.1	74.5	12.0	8.4
71.8	72.2	72.6	73.1	73.5	12.2	45.7	72.1	22.6	73.0	73.5	73.9	6.11	4.4
71.2	21.6	72.0	72.5	72.9	12.1	45.3	71.5	72.0	72.4	72.8	73.3	11.8	1.1
20.6	71.0	71.4	71.9	72.3	1,2,0	44.9	40.0	71.3	71.8	72.2	72.7	11.7	43.7

	1.379	18.0		48.9	49.4	20.0	50.5	51.1	51.7	52.2	52.8	53.3	53.9	54.4	55.0	55.0	20.1	56.7	57.2	57.8	58.3	58.9	59.4	0.00	90.0	01.1	61.7	02.7	02.8	93.3	93.9	4.4	65.0	9.5	1.99	66.7	67.3	8.29	68.3	
	1.376 74.0	17.9		49.3	49.7	50.3	20.8	51.4	52.0	52.5	53.1	53.0	54.2	54.7	55.3	55.9	56.4	57.0	57.5	58.1	58.7	59.2	29.8	60.3	60.0	01.5	62.0	02.0	93.I	03.7	64.2	64.8	65.4	62.0	66.5	67.0	9.29	68.2	68.7	
	1.373 73.5 Brix	17.8	Furity	49.4	<b>2</b> 0.0	20.6	S1.1	51.7	52.2	52.8	53.4	53.9	54.5	55.1	55.6	56.2	26.7	57.3	57.9	58.4	20.0	20.6	09.I	60.7	61.2	8.10	62.4	62.0	03.5	0.4°	04.0	65.2	65.7	66.3	6.99	67.4	08.0	68.5	1.69	
	1.371	17.7		49.7	50.3	50.8	51.4	52.0	52.5	53.1	53.7	54.2	54.8	55.4	55.9	50.5	57.1	27.6	58.3	58.8	59.3	59.9	60.5 60.5	61.0	9.19	62.1	62.7	63.3 ,	63.8	64.4 4.4	0.50	65.5	1.99	66.7	67.3	67.8	68.4	68.9	60 8	
	1.368	17.6		50.0	20.6	51.1	21.7	52.3	52.8	53.4	54.0	54.5	55.1	55.7	56.2	56.8	57.4	58.0	58.5	1.65	29.7	60.2	8.09	61.4	61.9	62.5	63.1	63.6	64.2	64.8	65.3	62.0	66.5	67.0	9.29	68.2	68.7	69.3	6.69	
wed.)	Sp. gr. of und. syp. Brix of und. syp.	t	Sug.	œ ·	8. O	9.0	9.1	9.2	6.3	4.6	ę.	9.6	7.6	о 89.	6.6	10.0	10,1	10.3	10.3	10.4	10.5	9.01	10.7	10.8	10.9	0.11	11.1	11.2	11.3	11.4	11.5	9'11	11.7	. i.	11.9	12.0	12.1	12.2	12.3	,
(Contin	Sp. gr. Brix o	,	P.K.	33.0	33.4	33.8	34.2	34.6	35.0	35.3	35.7	36.1	36.5	36.9	37.2	37.5	37.9	38.3	38.7	39.0	39.4	39.8	40.3	40.6	40.9	41.3	41.7	42.1	42.5	42.8	43.2	43.6	44.0	4.44	44.7	45.0	45.4	45.8	46.2	•
TABLE IV.—(Continued.)	1.365	17.5		1-64	46.7	50.3	20.0	51.4	52.0	\$2.6	53.1	53.7	54.3	54.9	55.4	56.0	26.6	57.1	57.7	58.3	58.9	59.4	0.09	9.09	61.1	61.7	62.3	62.9	63.4	64.0	64.6	65.1	65.7	99	66.9	67.4	68.0	9.89	1.69	1
T.	1.362	17.4		46.4	50.0	50.6	51.1	51.7	52.3	52.9	53.4	54.0	54.6	55.2	55.7	56.3	56.9	57.5	28.0	58.6	59.2	59.8	60.3	6.09	61.5	62.1	62.6	63.2	63.8	64.4	64.9	65.5	1.99	66.7	67.2	8.29	68.4	60.0	69.5	•
	1.359 71.3 Brix	17.3	Purity	49.7	50.3	50.9	51.4	52.0	52.6	53.2	53.8	54.3	54.9	55.5	26.1	26.6	57.2	57.8	58.4	59.0	59.5	60.1	60.7	61.3	8.19	62.4	63.0	63.6	64.2	64.7	65.3	62.0	99	67.1	9.79	68.2	889	69.4	60.69	
	1.357	17.2		50.0	50.6	\$1.2	51.7	52.3	52.9	53.5	54.1	54.7	55.2	55.0	56.4	57.0	57.6	58.1	58.7	59.3	59.9	60.5	61.0	9.19	62.2	62.8	63.4	64.0	64.5	65.1	65.7	66.3	66.0	67.4	68.0	989	60.2	60.8	70.3	
	1.354 70.5	17.1		50.3	50.0	51.5	52.0	52.6	53.2	53.8	54.4	55.0	55.6	56.1	56.7	57.3	57.0	200	59.1	59.6	60.2	80.8	61.4	62.0	62.6	63.2	63.7	64.3	64.9	65.5	1.99	66.7	67.3	67.8	68.4	60.0	90.0	70.2	20.8	
	Sp. gr. of und. syp. Brix of und. syp.		Sug.	8.6	8.7	.œ.	8.0	0.6	0.1	9.5	9.3	4.6	5.6	9.6	0.7	.00	0.0	10.0	10.1	10.2	10.3	10.4	10.5	9.01	10.7	10.8	10.9	11.0	11.1	11.2	11.3	11.4	11.4	11.6	11.7		0.11	12.0	12.1	•
	Sp. gr. Brix o		P.R.	32.2	32.6	33.0	33.4	33.7	34.1	34.5	34.0	35.2	35.6	36.0	36.4	9.9	37.3	37.5	37.0	38.3	38.7	30.0	30.3	30.7	40.1	40.5	8.04	41.2	41.6	42.0	42.4	42.8	43.1	43.5	43.0		? • •	4	45.4	

\$\frac{1}{2}\langle \frac{1}{2}\langle \frac{1}{2}\

	1.408	78.8	0.01	•	53.7	54.2	54.7	55.3	55.8	56.3	<b>26.8</b>	57.4	57.9	58.4	58.9	59.5	0.09	60.5	61.1	9.19	62.1	9.29	63.2	63.7	64.2	64.7	65.3	65.8	66.3	8.99	4.20	6.70	68.4	68.9	69.5	70.0	70.5	71.1	21.6	72.1	72.6
	1.40\$	78.4	18.0	ì	54.0	54.5	55.0	55.6	56.1	26.6	57.1	57.7	58.2	58.7	59.3	59.8	60.3	8.09	61.4	61.9	62.4	63.0	63.5	64.0	64.6	65.1	65.6	1.00	66.7	67.2	67.7	08.3 0	8.8	69.3	69.8 8.69	70.4	70.9	71.4	72.0	72.5	73.0
	1.402	77.9	Brix 18.8	Purity	54:3	54.8	55.3	55.9	56.4	26.9	57.4	28.0	58.5	59.0	29.6	1.09	9.09	61.2	61.7	62.2	62.8	63.3	63.8	64.4	64.9	65.4	99	66.5	67.0	9.29	1.6	9.80	1.69	69.7	70.3	70.7	71.3	71.8	72.3	72.9	73.4
	1.399	77.5	18.7	ì	54.5	55.1	55.6	56.2	26.7	57.2	57.8	58.3	58.8	59.4	59.0	<b>4.09</b>	0.19	61.5	62.0	62.6	63.1	63.6	64.2	64.7	65.2	65.8	66.3	90.8	67.4	6.29	68.4 4.8	0.60	69.5	70.1	20.6	71.1	7.1.7	72.2	72.7	73.3	73.8
	1.397	77.1	18.6		848	55.4	55.9	56.5	57.0	57.5	58.1	58.6	59.1	29.7	60.2	8.09	61.3	8.19	62.4	63.9	63.4	64.0	64.5	65.1	65.6	1.99	, 66.7	07.5	67.7	68.3	8.8	4.69	6.69	70.4	71.0	71.5	72.0	72.6	73.1	73.7	74.2
ed.)	of und. syp.	nd.		Sug.	10.2	10.3	10.4	10.5	9.01	10.7	10.8	6.01	0.11	1.1	11.2	11.3	11.4	11.5	9'11	11.7	8.11	6.11	12.0	12.1	12.3	12.3	12.4	12.5	12.6	12.7	12.8	12.9	13.0	13.1	13.2	13.3	13.4	13.5	13.6	13.7	13.8
IV. —(Continued.)	Sp. gr. of	Brix o		P.R.	38.4	38.8	39.3	39.6	40.0	40.4	40.7	41.1	41.5	41.9	42.3	42.7	43.0	43.3	43.7	44.1	44.5	44.9	45.2	45.6	46.0	46.4	46.7	47.1	47.5	47.9	8.3 6.3	48.0	49.0	49.4	49.8	50.2	50.5	50.9	51.3	51.7	52.0
TABLE IV.	1.393	9.92	8		54.1	54.6	55. I	55.7	56.2	56.8	57.3	27.8	58.4	58.9	59.5	0.0	60.5	61.1	9.19	62.2	62.7	63.2	63.8	64.3	64.9	65.4	65.0	90.5	67.0	92.0		9.80	69.2	69.7	70.3	20.8	71.4	71.9	72.4	73.0	78.5
17.	1.391	76.2	18.	•	54.3	54.9	55.4	56.0	56.5	57.1	27.6	58.2	58.7	59.5	26.8	60.3	6.09	61.4	62.0	62.5	63.0	63.6	64.1	64.7	65.2	65.8	66.3	90. 9	4.7	62.6	68.5	00.0	9.69	70.1	70.7	71.2	71.7	72.3	72.8	73.4	73.9
	1.388	75.8	Brix	Purity	54.6	55.2	55.7	56.3	56.8	57.4	57.9	58.5	59.0	29.6	60.1	60.7	61.2	61.7	62.3	62.8	63.4	63.0	64.5	65.0	65.6	1.99	66.7	67.2	67.8	98.3	68.9 (	9.4	6.69	70.5	71.0	9.1.6	72.1	72.7	73.2	73.8	74.3
	1.385	75.3	18.2		54.9	55.5	26.0	26.6	57.1	57.7	58.2	58.8	59.3	59.9	60.4	0.19	61.5	62.1	62.6	63.2	63.7	64.3	64.8	65.4	62.0	66.5	67.0	67.6	 	08.7	60,	8.60 8.00	70.3	70.9	71.4	72.0	72.5	73.1	73.6	74.2	74.7
	1.382	74.9	18.1		55.2	55.8	56.4	56.9	57.5	58.0	58.6	59.1	29.7	60.2	8.09	61.3	61.9	62.4	63.0	63.5	64.1	64.6	65.2	65.7	66.3	6.9	67.4	86.6 6.6		1.60	9.0	70.2	70.7	71.3	8.1.2	72.4	72.9	73.5	74.0	74.6	75.1
	Sp. gr. of und. syp.			Sug.	10.0	10.1	10.2	10.3	10.4	10.5	10.6	10.7	10.8	10.9	0.11	11.1	11.2	11.3	11.4	11.5	9.11	11.7	8.11	6.11	12.0	12.1	12.2	12.3	12.4	12.5	12.0	12.7	12.8	12.9	13.0	13.1	13.2	13.3	13.4	13.5	13.6
	Sp. gr.	Brix		P.R.	37.6	38.0	38.4	38.8	39.1	39.5	39.0	40.3	40.7	41.0	41.4	41.8	42.2	45.6	42.9	43.3	43.7	44.1	44.5	44.8	45.1	45.5	45.9	6.3	46.6	47.0	47.4	<b>4</b> 7.8	48.2	48.5	48.9	49.3	49.7	<b>5</b> 0.1	50.4	<b>5</b> 0.8	\$1.2

 $\begin{array}{c} 7.77 - 7.77 \\ 7.77 - 7.$ 

	1.438	83.3	20.0		54.0	54.5	55.0	55.5	26.0	56.5	57.0	57.5	58.0	58.5	29.0	59.5	0.09	60.5	61.0	61.5	62.0	62.5	63.0	63.5	04.0	64.5	65.0	5.5	°.	00.	o. 64.0	07.5	08.0	68.5	9.0	69.5	70.0	70.5	71.0	71.5	72.0
	1.435	82.8	10.0	•	54.3	54.8	55.3	55.8	56.3	56.8	57.3	57.8	58.3	58.8	59.3	29.8	60.3	8.09	61.3	61.8	62.3	62.8	63.3	63.8	04.3	64.8	65.3	65.8		8. 9.	67.3	67.8	88.3	68.8 8.8	69.3	8.69	70.4	70.9	71.4	71.9	72.4
	1.432	82.4	brix 10.8	Purity	54.5	55.1	55.6	56.1	26.6	57.1	27.6	58.1	58.6	59.1	29.6	1.09	9.09	61.1	9.19	62.1	62.6	63.1	63.6	64.1	9.4.0	65.2	65.7	66.2	66.7	67.2	67.7	68.2	68.7	69.3	2.69	70.2	70.7	71.2	711.7	72.2	72.7
	1.429	82.0	10.7		54.8 8	55.3	55.8	56.3	\$6.9	57.4	57.9	58.4	58.9	59.4	59.9	60.4	6.09	61.4	6.19	62.4	63.9	63.5	0.4.0	64.5	65.0	65.5	96.0		02.0	67.5	08.0	68.5	0.69 9	69.5	70.1	20.6	71.1	71.6	72.1	72.6	73.1
	1.427	81.6	10.6		55.1	55.6	26.1	26.6	57.1	57.7	58.2	58.7	59.2	29.7	60.2	60.7	61.2	61.7	62.2	62.8	63.3	63.8	64.3	64.8	65.3	65.8	99	8.00	67.3	62.6	4.89	68.0	69.4	6.69	70.4	70.9	71.4	71.9	72.4	73.0	73.5
ed.)	of und. syp.			Sug.	10.8	10.9	0.11	1.11	11.2	11.3	11.4	11.5	9.11	11.7	8.11	6.11	12.0	12.1	12.2	12.3	12.4	12.5	12.6	12.7	12.8	12.9	13.0	13.1	13.2	13.3	13.4	13.5	13.6	13.7	13.8	13.9	14.0	14.1	14.2	14.3	14.4
—(Continued.)	Sp. gr. of und.	Brix of		P.R.	40.9	41.3	41.6	42.0	42.4	42.8	43.2	43.5	43.9	44.3	44.7	45.1	45.4	45.8	46.2	46.6	47.0	47.3	47.7	48.1	48.5	48.8	49.2	49.6	20.0	50.4	50.7	51.1	51.5	51.9	52.3	52.6	53.0	53.4	53.7	54.2	54.5
TABLE IV.	1.423	81.1	10.8		53.8	54.4	54.9	55.4	55.9	56.4	56.9	57.4	57.9	58.5	59.0	59.5	0.09	60.5	61.0	61.5	62.1	62.6	63.1	63.6	64.1	9.49	65.1	65.6	66.2	2.99	67.2	67.7	68.3	68.7	69.5	69.7	70.3	20.8	71.3	71.8	72.3
£	1.420	90.6	10.4		54.1	54.6	55.2	55.7	56.2	26.7	57.2	57.7	58.2	58.8 8.8	59.3	59.8	60.3	8.09	61.3	61.9	62.4	62.9	63.4	63.9	64.4	64.9	65.5	0.09	66.5	67.0	67.5	0.89	9.89	1.69	9.69	70.1	20.6	71.1	71.6	72.2	72.7
	1.417	80.2	Brix 10.3	Purity	54.4	54.9	55.4	26.0	56.5	57.0	57.5	58.0	58.5	59.1	29.6	60.I	909	61.1	61.7	62.2	62.7	63.2	63.7	64.2	64.8	65.3	65.8 8.5.8	66.3	<b>6</b> 99	67.4	62.9	68.4	68.9	69.4	6.69	70.5	71.0	71.5	72.0	72.5	73.1
	1.414	29.8	10.2		54.7	55.2	55.7	56.2	56.8	57.3	57.8	58.3	58.9	59.4	59.9	60.4	6.09	61.5	62.0	62.5	63.0	63.5	64.1	64.6	65.1	65.6	66.1	66.7	67.2	67.7	68.2	68.7	69.3	8.69	70.3	70.8	71.4	71.9	72.4	72.9	73.4
	1.411	79.3	10.1		55.0	55.5	56.0	56.5	57.1	\$7.6	58.1	28.6	59.2	59.7	60.2	60.7	61.3	61.8	62.3	62.8	63.4	63.9	64.4	64.9	65.4	0.99	90.5	02.0	67.5	1.89	98.0	. 1.69	9.69	70.2	70.7	71.2	71.7	72.3	72.8	73.3	73.8
	syp.	syp.		ng.	ı'n	9	7	∞.	ó	o.	Ξ.	á		4	κċ	٠	.,	œ.	6.	0:	1:	7	€.	4	'n	۰	12.7	œ.	ė.	o.	-	Ŋ	8	4	'n	9	7	<b>œ</b>	6	o.	=
	Sp. gr. of und.	t und.		S	Ö	OI	o.	OI	01	1	I	=	II	11		I	Ξ	-	Ξ	7	ï	12	13	12	12	12	12	12	12	13	13	Ę	13.	13.	13	13.	13	Ę	ij	14	7

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## BEET-SUGAR MAKING

	1.469	9:	21.0		56.2	26.7	57.1	57.6	58.1	58.6	29.0	59.5	00.0	60.5	61.0	61.4	6.19	62.4	62.0	63.3	63.8	64.3	64.8	65.2	65.7	66.2	66.7	1.79	9.29	1.89	9.89	0.69	69.5	20.0	70.5	71.0	71.4	71.9	72.4	72.9	73.3
	1.466	?	20.9		56.5	56.9	57.4	57.9	58.4	58.8	59.3	29.8	60.3	8.09	61.2	61.7	62.2	62.7	63.2	63.6	64.1	64.6	65.1	65.5	0.99	66.5	67.0	67.5	62.9	4.8	6.89	69.4	6.69	70.3	70.8	71.3	71.8	72.2	72.7	73.2	73.7
	1.463	Brix	20.8	Purity	26.7	57.2	57.7	58.2	58.7	. 59. I	29.6	60.1	90.0	61.1	61.5	62.0	62.5	63.0	63.5	63.9	64.4	64.9	65.4	62.0	66.3	8.99	67.3	8.29	68.3	68.7	69.2	69.7	70.3	70.7	71.2	21.6	72.1	72.6	73.1	73.6	74.0
	1.460	6	20.7		57.0	57.5	58.0	58.5	58.9	59.4	59.9	60.4	600	61.4	61.8	62.3	62.8	63.3	63.8	64.3	64.7	65.2	65.7	66.2	66.7	67.1	9.29	1.89	9.89	1.69	9.69	70.0	70.5	71.0	71.5	72.0	72.5	72.9	73.4	73.9	74.4
	1.457	2	20.6		57.3	57.8	58.3	58.7	59.2	265	60.2	60.7	61.2	61.7	62.1	9.29	63.1	63.6	64.1	64.6	65.1	65.5	0.99	66.5	0.29	67.5	68.0	68.5	68.9	69.4	6.69	70.4	70.9	71.4	71.9	72.3	72.8	73.3	73.8	74.3	74.8
ued.)	Sp. gr. of und. syp.			Sug.	8.11	11.9	12.0	12.1	12.2	12.3	12.4	12.5	12.6	12.7	12.8	12.9	13.0	13.1	13.2	13.3	13.4	13.5	13.6	13.7	13.8	13.9	14.0	14.1	14.2	14.3	14:4	14.5	14.6	14.7	14.8	14.9	15.0	15.1	15.2	15.3	15.4
-(Continued.)	Sp. 81			P.R.	<b>4</b> .8	45.2	45.6	46.0	46.4	46.7	47.1	47.5	47.9	48.3	48.6	49.0	49.4	49.8	50.2	50.5	50.9	51.3	51.7	52.1	52.4	52.8	53.2	53.6	54.0	54.3	54.7	55.1	55.5	55.9	56.2	26.6	57.0	57.4	57.8	58.1	58.5
TABLE IV.	1.453	63:3	20.5		56.1	56.6	57.1	27.6	58.0	58.5	59.0	59.5	0.09	60.5	61.0	61.5	62.0	62.4	62.9	63.4	63.9	64.4	64.9	65.4	62.9	99	8.99	67.3	67.8	68.3	8.8	69.3	8.69	70.2	70.7	71.2	71.7	72.2	72.7	73.2	73.7
Ţ	1.451	1.60	20.4		56.4	56.9	57.4	57.8	58.3	58.8	59.3	<b>29.8</b>	60.3	8.09	61.3	61.8	62.3	62.7	63.2	63.7	64.2	64.7	65.2	65.7	66.2	2009	67.2	9.29	68.1	9.89	1.69	9.69	70.1	20.6	71.1	9.1.6	72.1	72.5	73.0	73.5	74.0
	1.448	Rri.	20.3	Purity	56.7	57.1	57.6	58.1	58.6	59.1	29.6	60.1	9.09	61.1	9.19	62.1	62.6	63.1	63.5	64.0	64.5	65.0	65.5	0.99	99.2	67.0	67.5	0.89	68.5	0.69	69.5	70.0	70.4	70.9	71.4	71.9	72.4	72.9	73.4	73.9	74.4
	1.445	÷	20.3		56.9	57.4	57.9	58.4	58.9	59.4	59.9	60.4	600	61.4	61.9	62.4	62.9	63.4	63.9	64.4	64.9	65.3	65.8	66.3	8.99	67.3	67.8	68.3	68.8	69.3	8.69	70.3	70.8	71.3	71.8	72.3	72.8	73.3	73.8	74.3	74.8
	1.442 8.8	2	20.1		57.2	57.7	58.2	58.7	59.2	59.7	60.2	60.7	61.2	61.7	62.3	62.7	63.2	63.7	64.2	64.7	65.2	65.7	66.2	66.7	67.2	67.7	68.3	68.7	69.2	69.7	70.1	20.6	71.1	9.1.6	72.1	72.6	73.1	73.6	74.1	74.6	75.1
	of und. syp.	į		Sug.	11.5	9.11	11.7	8.11	6.11	12.0	12.1	12.2	12.3	12.4	12.5	12.6	12.7	12.8	12.9	13.0	13.1	13.2	13.3	13.4	13.5	13.6	13.7	13.8	13.9	14.0	14.1	14.2	14.3	14.4	14.5	14.6	14.7	14.8	14.9	15.0	15.1
	Sp. gr. of	•		P.R.	43.6	44.0	44.4	4.8 8.4	45.1	45.5	45.9	46.3	46.7	47.0	47.4	47.8	48.2	48.6	48.9	49.3	49.7	50.1	50.5	50.8	51.2	51.6	52.0	52.4	52.7	53.1	53.5	53.9	54.3	54.6	55.0	55.4	55.7	20.3	26.5	<b>36.9</b>	57.3

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	1.503	92.4		22.0		58.2	28.6	20.1	59.5	0.09	60.5	6.09	61.4	61.8	62.3	62.7	63.2	63.6	64.1	64.5	65.0	65.5	62.9	66.4	8.99	67.3	67.7	68.2	98.0		9.5	70.0	70.5	70.9	71.4	71.8	72.3	72.7	73.2	73.6
	1.498	6.16		21.9		58.4	28.0	50.4	59.8	60.3	60.7	61.2	9.19	62.1	62.6	63.0	63.5	63.9	64.4	8.49	65.3	65.8	66.2	66.7	67.1	9.29	68.0	68.5	68.9	<b>4.6</b> 9	6.60	70.3	70.8	71.2	.71.7	72.1	72.6	73.1	73.5	74.0
	1.495	91.4	Brix	81.8	Purity	58.7	50.2	9.65	60.1	9.09	61.0	61.5	6.19	62.4	62.8	63.3	63.8	64.2	64.7	65.1	65.6	1.99	90.5	67.0	67.4	6.29	68.3	8.8	69.3	99.7	70.2	20.6	71.1	71.6	72.0	72.5	72.9	73.4	73.9	74.3
	1.492	91.0		21.7		60.0	50.4	20.0	60.4	8.09	61.3	8.19	62.2	62.7	63.1	63.6	64.1	64.5	65.0	65.4	62.9	4.99	8.99	67.3	67.7	68.3	68.7		9.0	70.0	70.5	71.0	71.4	71.9	72.4	72.8	73.3	73.7	74.2	74.7
	1.488	90.5		21.6		£0.3	50.7	60.2	9.09	61.1	9.19	62.0	62.5	63.0	63.4	63.9	64.4	64.8	65.3	65.7	66.2	66.7	67.1	9.29	68.1	68.5	0.69	69. 4.	6.60	70.4	70.0	71.3	71.8	72.2	72.7	73.1	73.6	74.1	74.5	75.0
wed.)	Sp. gr. of und. syp.	of und. syp.			Sug.	12.8	12.0	13.0	13.1	13.2	13.3	13.4	13.5	13.6	13.7	13.8	13.9	14.0	14.1	14.2	14.3	14.4	14.5	14.6	14.7	14.8	14.9	15.0	15.1	15.2	15.3	15.4	15.5	15.6	15.7	15.8	15.9	16.0	1.91	16.2
—(Continued.)	Sp. gr.	Brix			P.R.	48.8	49.2	49.6	50.0	50.4	50.8	51.1	51.5	51.9	52.3	52.7	53.0	53.4	53.8	54.2	54.6	54.9	55.3	55.7	56.1	56.5	56.8	57.2	27.0	0.0	50.4	58.8	59.1	59.5	89.9	60.3	60.7	61.1	61.5	61.9
TABLE IV.	1.485	90.0		21.5		67.7	28.	58.6	59.1	59.5	0.09	60.5	6.09	<b>61.4</b>	6.19	62.3	62.8	63.3	63.7	64.2	64.7	65.1	65.6	0.99	66.5	0.29	67.4	6.29	4.6	8.8	60,00	8.69	70.2	70.7	71.2	9.1.6	72.1	72.6	73.0	73.5
TA	1.482	89.6		21.4		67.0	58.4	58.0	59.3	59.8	60.3	2.09	61.2	61.7	62.1	62.6	63.1	63.6	64.0	64.5	65.0	65.4	62.0	66.4	8.99	67.3	67.8	68.2	08.7	60	0.60	70.1	20.6	71.0	71.5	72.0	72.4	72.9	73.4	73.8
	1.478	89.1	Brix	21.3	Purity	58.3	58.7	59.3	29.6	60.1	9.09	61.0	61.5	62.0	62.4	65.0	63.4	63.8	64.3	64.8	65.3	65.7	66.2	66.7	67.1	9.29	1.68	. S.	0.60	9.5	70.0	70.4	70.9	71.4	71.8	72.3	72.8	73.2	73.7	74.2
	1.476	88.7		21.2		58.5	59.0	59.4	59.9	60.4	8.0°	61.3	61.8 6	62.3	62.7	63.2	63.7	04.2	64.6	65.1	65.6	99	96.5	67.0	67.5	62.6	4.80	6.80	60.0	0.60	50	70.8	71.2	7.1.7	72.2	72.6	73.1	73.6	74.1	74.5
	1.473	88.3		21.1		58.8	59.3	29.7	60.2	60.7	1.10	o.r.o	02.1	02.0	03.0	93.5	04.0	04. S	9.50	65.4	62.0	66.4	8.99	67.3	8.29	68.7	29.7	2,00	60.7	10/	?	71.1	71.0	72.0	72.5	73.0	73.5	73.9	74.4	74.9
	Sp. gr. of und. syp.	of und. syp.			Sug.	12.4	12.5	12.0	12.7	12.8	12.9	13.0	13.1	13.2	13.3	13.4	13.5	13.0	13.7	13.8	13.9	14.0	14.1	14.2	14.3	14.4	24.5	0.1	÷;	0.4.		15.0	15.1	15.2	15.3	15.4	15.5	15.6	15.7	15.8
1	Sp. gr.	Brix		1	P.R.	47.2	47.6	0.0	40.4	40.7	1.04	45.5 5	4 r 2 c	5	20.0	9	51.4	51.0	52.2	52.5	52.0	53.3	53.7	54.1		54.0	20.0	20.0	2 4	4 4			57.5	57.9	, S	58.0	20.0	59.4	20.0	2.00

3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 + 6.576 = 9.000 3.576 = 9.0000 3.576 = 9.0000 3.576 = 9.0000 3.576 = 9 $\begin{array}{c} 457777\\ 461.07$ 

	1.535	6.96		23.0		60.09	61.3	61.7	62.2	9.29	63.0	03.5	63.9	04.3	64.8	65.2	65.7	1.99	99.3	67.0	67.4	67.8	68.3	68.7	1.69	9.69	20.0	70.4	70.9	71.3	71.7	72.2	72.6	73.0	73.5	73.9	74.3	74.8	75.2	78.7
	1.531	96.4		22.9		61.1	91.9	62.0	62.4	62.9	63.3	63.8	64.2	0.4.0	65.1	65.5	62.0	66.4	8.99	67.3	67.7	1.89	9.89	0.69	4.69	669	70.3	70.7	71.2	21.6	72.1	72.5	72.9	73.4	73.8	74.2	74.7	75.1	75.5	26.0
	1.528	96.0	Brix	22.8	Purity	61.4	61.8	62.3	62.7	63.2	63.6	04.0	64.5	64.9	65.4	65.8	66.2	299	67.1	67.5	68.0	68.4	68.0	69.3	69.7	70.2	20.6	71.1	71.5	71.9	72.4	72.8	73.2	73.7	74.1	74.6	75.0	75.4	75.9	2.92
	1.525	95.5		22.7		61.7	62.1	62.6	63.0	63.4	63.9	64.3	64.8	65.2	65.6	1.99	66.5	67.0	67.4	.8.49	68.3	68.7	69.2	9.69	70.0	70.5	70.9	71.4	71.8	72.2	72.7	73.1	73.6	74.0	74.4	74.9	75.3	75.8	76.2	76.7
	1.522	95.1		22.6		61.9	62.4	62.8	63.3	63.7	64.2	04.0	65.0	65.5	62.9	66.4	8.99	67.3	67.7	68.1	9.89	0.69	69.5	6.69	70.4	70.8	71.2	711.7	72.1	72.6	73.0	73.5	73.9	74.3	74.8	75.2	75.7	76.1	76.5	77.0
med.)	Sp. gr. of und. syp.	of und. syp.			Sug.	14.0	14.1	14.2	14.3	14.4	14.5	14.6	14.7	14.8	14.9	15.0	15.1	15.2	15.3	15.4	15.5	15.6	15.7	15.8	15.9	0.91	191	16.2	16.3	16.4	16.5	9.91	16.7	16.8	16.9	17.0	17.1	17.2	17.3	17.4
. — (Continued.	Sp. gr.	Brix of			P.R.	53.6	54.0	54.4	54.8	55.2	55.6	55.9	56.3	20.7	57.1	57.5	57.9	58.2	58.6	29.0	59.4	29.8	60.2	60.5	6009	61.3	61.7	62. I	62.4	62.8	63.2	63.6	04.0	64.3	64.7	65.1	65.5	62.9	66.3	2.99
LABLE IV	1.518	94.6		22.5		59.1	29.6	0.09	60.4	6.09	61.3	8.10	62.2	62.7	63.1	63.6	0.40	64.4	64.9	65.3	65.8	66.2	66.7	1.29	9.29	68.0	68.4	68.9	69.3	8.69	70.2	70.7	71.1	71.6	72.0	72.4	72.9	73.3	73.8	74.3
H	1.515	94.2		22.4		59.4	8.65	60.3	60.7	61.2	9.19	62.1	62.5	62.0	63.4	63.8	64.3	64.7	65.2	65.6	1.99	66.5	62.0	67.4	62.9	68.3	68.7	69.2	9.69	70.1	70.5	71.0	71.4	71.9	72.3	72.8	73.2	73.7	74.1	24.6
	1.511	93.7	Brix	22.3	Purity	59.6	1.09	60.5	0.19	61.4	61.9	62.3	62.8	63.2	63.7	64. I	64.6	65.0	65.5	62.0	66.4	8.99	67.3	67.7	68.2	9.89	1.69	69.5	70.0	70.4	70.9	71.3	71.7	72.3	72.6	73.1	73.5	74.0	74.4	74.0
	1.508	93.2		22.2		59.9	60.4	8.09	61.3	61.7	62.2	02.0	63.1	63.5	0.40	64.4	64.9	65.3	65.8	66.2	66.7	67.1	9.29	e8.0	68.5	68.9	69.4	. <b>69.</b> 8	70.3	7.07	71.2	9.1.6	72.1	72.5	73.0	73.4	73.9	74.3	74.8	75.2
	1.505	92.8		<b>3</b> 2.1		60.2	9.09	61.1	61.5	62.0	62.4	62.0	63.3	63.8	64.3	64.7	65.2	65.6	1.99	66.5	67.0	67.4	62.9	68.3	8.8	69.2	69.7	70.1	20.6	71.0	71.5	71.9	72.4	72.9	73.3	73.8	74.2	74.7	75.1	75.6
	Sp. gr. of und. syp.	of und. syp.			Sug.	13.3	13.4	13.5	13.6	13.7	13.8	13.9	14.0	14.1	14.2	14.3	14.4	14.5	14.6	14.7	14.8	14.9	15.0	13.1	15.2	15.3	15.4	15.5	15.6	15.7	15.8	15.9	16.0	1.6.1	16.2	16.3	16.4	16.5	16.6	16.7
	Sp. g	Brix of			P.R.	50.8	51.2	\$1.6	52.0	52.4	52.8	53.2	53.5	53.9	54.3	54.7	55.1	55.4	55.8	56.2	56.6	57.0	57.4	57.8	58.2	58.6	58.9	59.3	26.4	60.0	60.4	80.8	61.2	61.6	61.9	62.3	62.7	63.1	63.5	0.29

TABLES 331

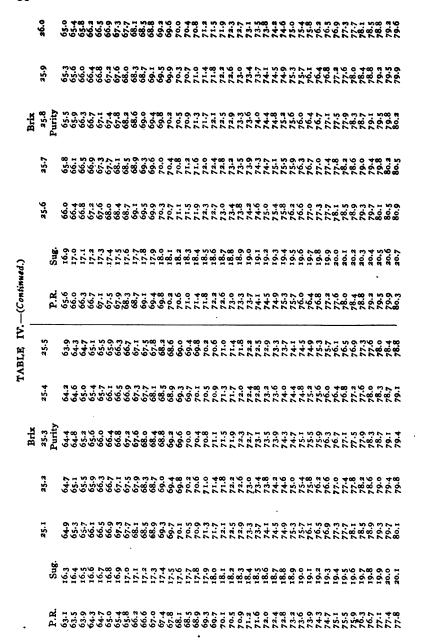
76.1	26.5	77.0	77.4	77.8	78.3	78.7	79.1	29.6	80.0	80.4	80.0	81.3	81.7	82.2	82.6	83.0	83.5	83.9	84.3	84.8	85.2	85.7	86.1	86.5	87.0	87.4	87.8	88.3	88.7	89.1	89.6	0.06	90.4	6.06	91.3	91.7	92.2	92.6	93.0	93.5	93.9	94.3	94.8
76.4	76.9	77.3	77.7	78.2	78.6	79.0	79.5	79.9	80.3	80.8	81.2	81.7	82.1	82.5	83.0	83.4	83.8	84.3	84.7	85.2	85.6	86.0	86.5	86.9	87.3	87.8	88.2	88.6	89.1	89.5	0.06	90.4	90.8	91.3	7.16	92.1	95.6	93.0	93.4	93.9	94.3	94.8	95.2
26.8	77.2	27.6	78.1	78.5	78.9	79.4	8.62	80.3	80.7	81.1	81.6	82.0	82.5	82.9	83.3	83.8	84.2	84.6	85.1	85.5	86.0	86.4	8.98	87.3	87.7	88.2	88.6	89.0	89.5	89.9	90.4	8.06	91.2	91.7	92.1	92.5	93.0	93.4	93.9	94.3	94.7	95.2	93.0
77.1	77.5	78.0	78.4	78.9	79.3	79.7	80.7	80.6	81.1	81.5	81.9	82.4	82.8	83.3	83.7	84.1	84.6	85.0	85.5	85.9	86.3	86.8	87.3	87.7	88.1	88.5	89.0	89.4	89.9	90.3	8.00	91.2	916	92.1	92.5	93.0	93.4	93.8	94.3	94.7	95.2	95.0	96.0
77.4	77.9	78.3	78.8	79.2	79.6	80.1	80.5	81.0	81.4	81.9	82.3	82.7	83.2	83.6	84.1	84.5	85.0	85.4	85.8	86.3	86.7	87.2	87.6	88.1	88.5	88.0	89.4	89.8	90.3	90.7	91.2	91.6	92.0	92.5	92.9	93.4	93.8	94.2	94.7	95.1	95.6	96.0	96.5
																									20.0			•	.,	-	•	•			•	.,					-		7
67.1	67.5	6.79	68.2	9.89	69.0	69.4	69.8	70.1	70.5	70.9	71.3	71.7	72.0	72.4	72.8	73.2	73.6	73.9	74.3	74.7	75.1	75.5	75.8	76.3	29.9	77.0	77.4	77.8	78.2	78.6	79.0	79.4	79.7	80.1	80.5	80.9	81.3	9.18	82.0	82.4	82.8	83.2	83.5
74.7	75.1	75.6	26.0	76.4	76.9	77.3	77.8	78.3	78.7	79.1	29.6	80.0	80.4	80.9	81.3	81.8	82.2	82.7	83.1	83.6	84.0	84.4	84.9	85.3	85.8	86.2	86.7	87.1	87.6	88.0	88.4	88.0	89.3	89.8	90.3	2006	91.1	9.16	92.0	92.4	92.9	93.3	93.8
75.0	75.4	75.9	76.3	76.8	77.2	77.7	78.1	78.6	79.0	79.5	79.9	80.4	80.8	81.2	81.7	82.1	82.6	83.0	83.5	83.9	84.4	84.8	85.3	85.7	86.2	9.98	87.1	87.5	87.9	88.4	88.8	89.3	89.7	90.2	90.6	1.16	91.5	92.0	92.4	92.9	93.3	93.7	94.2
75.3	75.8	76.3	76.7	77.1	27.6	78.0	78.5	78.9	79.4	79.8	80.3	80.7	81.2	81.6	82.1	82.5	83.0	83.4	83.9	84.3	84.8	85.2	85.7	86.1	86.5	87.0	87.4	87.9	88.3	88.8	89.2	89.7	1.06	90.0	91.0	91.5	91.9	92.4	92.8	93.3	93.7	94.2	94.0
75.7	76.1	26.6	77.0	77.5	77.9	78.4	78.8	79.3	79.7	80.2	80.6	81.1	81.5	82.0	82.4	82.9	83.3	83.8	84.2	84.7	85.1	85.6	86.0	86.5	86.9	87.4	87.8	88.3	88.7	89.3	89.6	1.06	90.5	91.0	91.4	91.9	92.3	92.8	93.2	93.7	94.1	94.0	95.0
26.0	76.5	76.9	77.4	77.8	78.3	78.7	79.2	79.6	80.1	80.5	81.0	81.4	81.9	82.4	82.8	83.3	83.7	84.2	84.6	85.1	85.5	86.0	86.4	86.9	87.3	87.8	88.3	88.7	89.1	89.6	90.0	90.5	91.0	91.4	6.16	92.3	92.8	93.2	93.7	94.1	94.6	95.0	95.5
16.8	16.9	17.0	17.1	17.3	17.3	17.4	17.5	17.6	17.7	17.8	17.9	18.0	18.1	18.3	18.3	18.4	18.5	18.6	18.7	18.8	18.9	19.0	1.61	19.2	19.3	19.4	19.5	19.6	19.7	19.8	19.9	20.0	20.I	20.2	20.3	20.4	20.5	20.6	20.7	20.8	20.0	21.0	21.1
64.3	64.6	65.0	65.4	65.8	99	9	6.9	67.3	67.7	88.	68.4	8.8	69.7	9.6	6,69	70.3	70.7	71.1	71.5	71.8	72.2	72.6	73.0	73.4	73.8	74.2	74.6	75.0	75.4	75.7			26.9	77.3	27.6	8.0	78.4	8.8	79.2	79.5	79.9	œ.	80.7

	•		24.0		8.09	61.2	61.7	02.1	62.5	63.0	03.3	03.7	4.2	0.4.0	65.0	65.4	65.8	66.2	66.7	67.1	67.5	67.9		68.7	60.2	9.69	70.0	70.4	20.8	71.3	71.7	72.1	72.5	72.9	73.3	73.7	74.2	74.6	75.0	75.4
			23.9		61.1	61.5	61.9	62.3	62.8	63.2	93.0	0.4.0	4.4	04.9	65.3	65.7	1.00	<b>9</b> 0.5	6.99	67.4	67.8	68.2	98.0	0.69	69.5	60.0	70.3	70.7	71.1	71.5	72.0	72.4	72.8	73.2	73.6	74.1	74.5	74.9	75.3	75.7
		Brix	23.8	Purity	61.3	8.19	62.2	62.6	63.0	63.4	63.0	64.3 8	64.7	65.1	65.5	0.9	66.4	<b>8.99</b>	67.3	9.29	1.8	68.5	68.9	69.3	69.7	70.2	20.6	71.0	71.4	71.8	72.3	72.7	73.1	73.5	73.9	74.4	74.8	75.2	75.6	76.1
			23.7		9.19	62.0	62.4	62.0	63.3	63.7	1.40	04.0	02.0	65.4	65.8	66.2	66.7	67.1	67.5	62.9	68.4	8.8	69.2	9.69	20.0	70.5	70.9	71.3	71.7	72.2	72.6	73.0	73.4	73.8	74.3	74.7	75.1	75.5	75.9	76.4
			23.6		6.19	62.3	62.7	63.1	63.6	0.4.0	64.4	8.4.8	65.3	65.7	1.99	66.5	6.9	67.4	67.8	68.3	9.89	69.1	69.5	6.69	70.3	70.8	71.2	9.1.6	72.0	72.5	72.9	73.3	73.7	74.2	74.6	75.0	75.4	75.8	76.3	76.7
ed.)				Sug.	14.6	14.7	14.8	14.9	15.0	15.1	15.2	15.3	15.4	15.5	15.6	15.7	15.8	15.9	16.0	1.91	16.2	16.3	16.4	16.5	16.6	16.7	16.8	16.9	17.0	17.1	17.2	17.3	17.4	17.5	17.6	17.7	17.8	17.9	18.0	18.1
-(Continu				P.R.	56.2	26.6	56.9	57.3	57.7	58.1	58.5	68.9	59.3	29.6	0.09	60.4	8.09	61.2	9.19	62.0	62.3	62.7	63.0	63.4	63.8	64.2	64.6	65.0	65.4	65.8	66.2	9.99	67.0	67.3	67.7	68.1	68.5	68.9	69.3	69.7
TABLE IV.—(Continued.)	1.552	60.7	23.5	;	0.09	60.4	0.09	61.3	61.7	62.1	62.6	63.0	63.4	63.8	64.3	64.7	65.1	65.5	0.99	99	8.99	67.3	67.7	68.1	68.5	689	69.4	8.69	70.3	20.6	71.1	71.5	71.9	72.3	72.8	73.2	73.6	74.0	74.5	74.9
TA	1.549	8.86 8.8	23.4		60.1	60.7	61.1	61.5	62.0	62.4	62.8	63.2	63.7	64.1	64.5	65.0	65.4	8.59	66.2	66.7	67.1	67.5	62.0	68.4	68.8	69.3	69.7	70.1	70.5	70.9	71.4	71.8	72.2	72.6	73.1	73.5	73.9	74.4	74.8	75.2
	1.545	98.3 Brix	23.3	Purity	60.5	60.0	61.4	61.8	62.2	62.7	63.1	63.5	63.0	64.4	64.8	65.2	65.7	66.1	₹99	62.0	67.4	67.8	68.2	68.7	69.1	69.5	70.0	70.4	70.8	71.2	71.7	72.1	72.5	73.0	73.4	73.8	74.2	74-7	75.1	75-5
	1.542	6.76	23.2		8.09	61.2	9.19	62.1	62.5	62.0	63.4	63.8	64.2	64.7	65.1	65.5	62.0	66.4	8.99	67.2	67.7	1.89	68.5	0.69	69.4	8. 69	70.3	70.7	71.1	71.6	72.0	72.4	72.8	73.3	73.7	74.1	74.6	75.0	75.4	75.9
	1.538	97.4	23.1		61.0	61.5	61.9	62.3	62.8	63.2	63.6	64.1	64.5	64.9	65.4	65.8	66.3	66.7	67.1	67.5	68.0	68.4	8.8	69.3	69.7	70.1	9.02	71.0	71.4	71.9	72.3	72.7	73.2	73.6	74.0	74.5	74.9	75.3	75.8	76.2
	und. syp.	und. syp.		Sug.	14.1	14.2	14.3	14.4	14.5	14.6	14.7	14.8	14.9	15.0	15.1	15.2	15.3	15.4	15.5	15.6	15.7	15.8	15.9	16.0	1.91	16.2	16.3	16.4	16.5	16.6	16.7	16.8	16.9	17.0	17.1	17.2	17.3	17.4	17.5	17.6
	Sp. gr. of und. s	DIX OF		P.R.	54.1	54.5	54.9	54.3	55.7	56.1	56.5	56.8	57.2	27.6	58.0	58.4	58.8	59.1	59.5	59.0	60.3	60.7	61.1	61.4	61.8	62.2	62.6	63.0	63.4	63.8	64.2	64.5	64.9	65.3	65.7	0.99	66.4	8.99	67.3	9.29

\$277.7 \$4.00.7 \$1.00.7 

		25.0		62.8	63.2	03.0 03.0	<b>6</b> 4.0	4.4	8.4.8	65.2	9.5	99	66.4	8.9	67.2	9.29	08.0	68.4	68.8	69.7	9.69	20.0	70.4	70.8	71.2	21.6	72.0	72.4	73.8	73.5	73.0	74.0	74.4	74.8	75.3	75.0	20.0	76.4	76.8	77.3	27.6	78.0
		24.9		63.1	63.5	63.0	64.3	04.7	65.1	65.5	6.5	66.3	66.7	67.1	67.5	62.0	68.3	68.7	1.69	69.5	6.6 6	70.3	70.7	71.1	71.5	6.17	72.3	72.7	73.1	73.5	73.9	74:3	74.7	75.1	75.5	75.9	76.3	76.7	77.1	77.5	77.9	78.3
	Brix	24.8	Purity	63.3	63.7	1.40	64.5	04.0	65.3	65.7	1.09	96.5	6.99	67.3	67.7	98.1	68.5	68.9	69.4	8.69	70.2	20.6	71.0	71.4	71.8	72.2	72.6	73.0	73.4	73.8	74.2	74.0	75.0	75.4	75.8	76.2	9.92	77.0	77.4	77.8	78.2	78.6
		24.7		63.6	04.0	4.4	64.8	65.2	65.6	0.0	66.4	8.9	67.2	9.29	80	68.4	8.8	69.2	9.69	20.0	70.4	70.9	71.3	7.1.7	72.1	72.5	72.9	73.3	73.7	74.1	74.5	74.9	75.3	75.7	70.1	76.5	26.9	77.3	7.7.7	78.1	78.5	78.9
		34.6		63.8	64.2	0 <del>.</del> 0	6 <b>5</b> .0	65.4	65.9	66.3	66.7	67.1	67.5	62.9	68.3	68.7	1.69	69.5	6.69	70.3	70.7	71.1	71.5	71.9	72.4	72.8	73.2	73.6	74.0	4.4	24.8	75.2	75.0	76.0	76.4	26.8	77.3	27.6	78.0	78.5	78.9	79.3
ed.)			Sug.	15.7	15.8	15.9	16.0	1.01	16.2	16.3	16.4	16.5	9.91	16.7	16.8	6.9	17.0	17.1	17.2	17.3	17.4	17.5	17.6	17.7	17.8	6.21	0.6	 	18.2	18.3	18.4	18 2.5	18.0	18.7	18.00 0.00	18.9	19.0	1.61	19.2	19.3	19.4	19.5
IV (Continued.)			P.R.	60.7	61.1	4:10	8.19	62.2	62.6	63.0	63.4	63.8	64.2	9.4.0	64.9	65.3	65.7	1.99	66.5	8.99	67.3	9.29	68.o	68.4	68.8	69.2	9.69	6.69	70.3	70.7	71.1	71.5	71.9	72.3	72.6	73.0	73.4	73.8	74.2	74.6	75.0	75.3
TABLE IV.		24.5		62.0	62.4	62.0	63.3	63.7	1.49	64.5	64.9	65.3	65.7	1.99	66.5	6.99	67.3	8.29	68.3	9.89	0.69	69.4	8.69	70.2	20.6	71.0	71.4	71.8	72.2	72.7	73.1	73.5	73.9	74.3	74.7	75.1	75.5	75.9	76.3	76.7	77.1	77.6
ΤA		24.4		62.3	62.7	63.1	63.5	63.9	64.3	64.8	65.2	65.6	0.99	66.4	8.99	67.2	9.29	68.0	68.4	689	69.3	69.7	70.1	70.5	70.9	71.3	711.7	72.1	72.5	73.0	73.4	73.8	74.2	74.6	75.0	75.4	75.8	76.2	26.6	77.0	77.5	77.9
	Brix	24.3	Purity	62.6	63.0	63.4	63.8	64.2	64.6	65.0	65.4	65.8	66.3	299	67.1	67.5	62.9	68.3	68.7	1.69	69.5	70.0	70.4	70.8	71.2	71.6	72.0	72.4	72.8	73.3	73.7	74.1	74.5	74.9	75.3	75.7	76.1	76.5	77.0	77.4	27.8	78.2
		24.2		62.8	63.2	63.6	0.40	64.5	64.9	65.3	65.7	1.99	66.5	6.99	67.4	67.8	68.2	9.89	0.69	69.4	8.69	70.2	70.7	71.1	71.5	71.9	72.3	72.7	73.1	73.6	74.0	74.4	74.8	75.2	75.6	26.0	76.4	69/	77.3	77.7	78.1	78.5
		24·I		63.1	63.5	63.9	64.3	64.7	65.1	65.6	0.99	66.4	8.99	67.2	9.29	68.0	68.5	689	69.3	69.7	70.1	70.5	71.0	71.4	71.8	72.2	72.6	73.0	73.4	73.9	74-3	74.7	75.1	75.5	75.9	76.3	76.8	77.2	77.6	78.0	78.4	78.8
			Sug.	15.2	15.3	15.4	15.5	15.6	15.7	15.8	15.9	16.0	16.1	16.2	16.3	16.4	16.5	16.6	16.7	16.8	16.9	17.0	17.1	17.2	17.3	17.4	17.5	17.6	17.7	17.8	17.9	18.0	18.1	18.2	18.3	18.4	18.5	18.6	18.7	8.8	18.9	0.61
			P.R.	58.6	59.0	59.4	59.8	60.2	60.5	6.09	61.3	61.7	62.1	62.5	65.9	63.3	63.7	04.0	64.4	8.49	65.2	65.6	0.99	66.3	2.99	67.1	67.5	62.9	68.3	9.89	0.69	69.4	8.69	70.2	20.6	71.0	71.3	71.7	72.1	72.5	72.9	73.3

78.4	78.8	79.7	20.6	80.0	80.4	80.8	81.2	81.6	82.0	82.4	82.8	83.2	83.6	<b>8</b>	84.4	84.8	85.2	85.6	86.0 8	86.4	86.8	87.2	87.6	<b>8</b> 80.0	88.4	88.8	89.2	89.6	90.0	90.4	90.8	91.2	91.6	92.0	92.4	92.8	93.2	93.6	94.0
78.7	1.67	79.5	6.64	80.3	80.7	81.1	81.5	81.9	82.3	82.7	83.1	83.5	83.9	84.3	84.7	85.1	85.5	85.9	86.3	86.7	87.1	87.6	88.0	88.4	88.8	89.2	89.6	90.0	90.4	90.8	91.2	9.16	92.0	92.4	92.8	93.2	93.6	94.0	94.4
29.0	79.4	26.8	80.2	80.6	81.0	81.5	81.9	82.3	82.7	83.1	83.5	83.9	84.3	84.7	85.1	85.5	85.9	86.3	86.7	87.1	87.5	87.9	88.3	88.7	1.68	89.5	89.9	90.3	2.06	1.16	91.5	6.16	92.3	92.7	93.1	93.5	94.0	94.4	94.8
79.4	8.62	80.2	80.6	81.0	81.4	81.8	82.3	82.6	83.0	83.4	83.8	84.2	84.6	85.0	85.4	85.8	86.2	9.98	87.0	87.4	87.9	88.3	88.7	89.1	89.5	89.9	90.3	90.7	91.1	91.5	91.9	92.3	92.7	93.1	93.5	93.9	94.3	94.7	95.1
79.7	80.1	80.5	80.9	81.3	81.7	82.1	82.5	82.9	83.3	83.7	84.1	84.6	85.0	85.4	85.8	86.2	9.98	87.0	87.4	87.8	88.2	88.6	89.0	89.4	86.8	90.2	2005	1.16	91.5	91.9	92.3	92.7	93.1	93.5	93.9	94.3	94.7	95.1	95.5
19.6	19.7	19.8	19.9	20.0	20. I	20.2	20.3	20.4	20.5	20.6	20.7	8.02	20.9	21.0	21.1	21.3	21.3	21.4	21.5	21.6	21.7	21.8	21.9	22.0	22.1	22.2	22.3	22.4	22.5	22.6	22.7	22.8	22.9	23.0	23.1	23.2	23.3	23.4	23.5
75.7	76.1	76.5	6.97	77.3	77.7	78.1	78.5	78.8	79.2	29.6	80.0	80.4	80.8	81.1	81.5	81.9	82.3	82.7	83.1	83.5	83.9	84.2	84.6	85.0	85.4	85.8	86.2	86.5	86.9	87.3	87.7	88.1	88.5	88.0	89.3	89.6	90.0	90.4	8.06
78.0	78.4	78.8	79.3	9.6	80.0	80.4	80.8	81.2	91.6	82.0	82.4	82.9	83.3	83.7	84.1	84.5	84.9	85.3	85.7	1.98	86.5	86.9	87.3	87.8	88.2	88.6	89.0	89.4	89.8	90.2	9.06	91.0	91.4	91.8	92.2	92.7	93.1	93.5	93.9
78.3	78.7	1.62	79.5	29.9	80.3	80.7	81.1	81.6	82.0	82.4	83.8	83.2	83.6	84.0	84.4	84.8	85.2	85.7	86.1	86.5	86.9	87.3	87.7	88.1	88.5	88.9	89.3	89.8	90.2	90.6	91.0	91.4	8.16	92.3	93.6	93.0	93.4	93.9	94.3
78.6	79.0	79.4	8.62	80.2	80.7	81.1	81.5	81.9	82.3	82.7	83.1	83.5	84.0	84.4	84.8	85.2	85.6	86.0	86.4	86.8	87.2	87.7	88.1	88.5	88.0	89.3	89.7	1.06	90.5	6.06	91.4	91.8	92.2	93.6	93.0	93.4	93.8	94.2	94.7
78.9	79.3	29.8	80.2	80.6	81.0	81.4	81.8	82.2	82.6	83.1	83.5	83.9	84.3	84.7	85.1	85.5	86.0	86.4	86.8	87.2	87.6	88.0	88.4	88.8	89.3	89.7	1.06	90.5	6.06	91.3	61.7	92.1	95.6	93.0	93.4	93.8	94.2	94.6	95.0
79.3	79.7	80.1	80.5	80.9	81.3	81.7	82.2	82.6	83.0	83.4	83.8	84.2	84.6	85.1	85.5	85.9	86.3	86.7	87.1	87.6	88.0	88.4	88.8	89.3	89.6	0.06	90.5	90.0	91.3	7.16	92.1	92.5	92.9	93.4	93.8	94.2	94.6	95.0	95.4
1.61	19.2	19.3	19.4	19.5	19.6	19.7	8.61	19.9	20.0	20.1	20.2	20.3	20.4	20.5	20.6	20.7	20.8	20.9	21.0	21.1	21.2	21.3	21.4	21.5	21.6	21.7	21.8	21.9	22.0	22.1	22.2	22.3	22.4	22.5	22.6	22.7	22.8	22.9	23.0
3.7	. <del>.</del>	4.4	8.	5.2	5.6	6.0	6.3	6.7	7.1	7.5	6.2		8.7	0.6	9.4	8.	0.2	9.0	0.1	1.4	1.7	2.1	2.5	2.0		3.7	0.4	4.4	8.4	5.2	5.6	0.0	6.4	6.8	7.1	7.5	7.9	 	8.7



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	27.0	•	62.0	67.4	67.8	68.1	68.5	68.9	69.3	9.69	70.0	70.4	70.7	71.1	71.5	71.9	72.3	72.6	73.0	73.3	73.7	74.1	74.4	74.8	75.2	75.6	75.9	76.3	76.7	77.0	77.4	77.8	78.1	78.5	78.9	79.3	29.6	80.0	80.4	80.7	81.1	
	26.9	,	67.3	67.7	68.0	68.4	68.8	1.69	69.5	6.69	70.3	20.6	71.0	71.4	71.7	72.1	72.5	72.9	73.2	73.6	74.0	74.3	74.7	75.1	75.5	75.8	76.2	9.92	77.0	77.3	7.7.7	78.1	78.4	7 <b>8</b> .8	79.2	29.6	79.9	80.3	80.7	81.0	81.4	
	Brix 26.8	Purity	67.5	62.9	68.3	68.7	0.69	<b>%</b>	8.69	70.1	70.5	50.0	71.3	71.6	72.0	72.4	72.8	73.1	73.5	73.9	74.3	74.6	75.0	75.4	75.7	76.1	76.5	6.9	77.2	27.6	78.0	78.4	78.7	1.62	79.5	29.9	80.2	80.6	81.0	81.3	81.7	
	26.7		8.79	68.2	68.5	6.89	69.3	69.7	70.0	70.4	70.8	71.2	71.5	71.9	72.3	72.7	73.0	73.4	73.8	74.2	74.5	74.9	75.3	75.7	26.0	76.4	76.8	77.2	77.5	77.9	78.3	78.7	29.0	79.4	8.64	80.2	80.5	80.9	81.3	81.7	82,0	
	36.6		68.0	68.4	8.89	69.2	69.5	6.69	70.3	70.7	71.1	71.4	71.8	72.2	72.6	72.9	73.3	73.7	74.1	74.4	74.8	75.2	75.6	75.9	76.3	76.7	77.1	77.4	77.8	78.7	78.6	78.9	79.3	79.7	80.1	80.5	80.8	81.2	81.6	82.0	82.3	
rd.)		Sug.	18.1	18.2	18.3	18.4	18.5	18.6	18.7	18.8	18.9	0.61	19.1	19.2	19.3	19.4	19.5	9.61	19.7	8.61	19.0	20.0	20.I	20.2	20.3	20.4	20.5	20.6	20.7	20.8	20.9	21.0	21.1	21.2	21.3	21.4	21.5	21.6	21.7	21.8	21.9	
—(Continued.)		P.R.	70.5	70.9	71.3	71.7	72.1	72.5	72.9	73.3	73.6	74.0	74.4	74.8	75.2	75.6	26.0	76.4	76.8	77.2	77.5	77.9	78.3	78.7	79.1	79.5	6.62	80.3	80.7	81.1	81.4	81.8	82.2	82.6	83.0	83.4	83.8	84.2	84.6	85.0	85.3	
LABLE IV	26.5	,	0.99	66.4	8.99	67.2	67.5	62.9	68.3	68.7	1.69	4.69	8.69	70.2	20.0	70.9	71.3	7x.7	72.1	72.5	72.8	73.2	73.6	74.0	74.3	74.7	75.1	75.5	75.8	76.2	26.6	27.0	77.4	77.7	78.1	78.5	78.9	79.2	9.6	80.0	80.4	
TAE	26.4	•	66.3	2.99	67.0	67.4	67.8	68.2	9.89	68.9	69.3	2.69	70.1	70.5	70.8	71.2	9.1.6	72.0	72.3	72.7	73.1	73.5	73.9	74.2	74.6	75.0	75.4	75.8	76.1	76.5	76.9	77.3	77.7	78.0	78.4	78.8	79.2	79.5	6.64	80.3	80.7	
	Brix 26.3	urity	66.5	6.9	67.3	67.7	68.1	68.4	8.8	69.2	9.69	70.0	70.3	70.7	71.1	71.5	71.9	72.2	72.6	73.0	73.4	73.8	74.1	74.5	74.9	75.3	75.7	76.0	76.4	76.8	77.2	77.0	77.9	78.3	78.7	1.62	79.5	8.62	80.2	80.6	81.0	
	26.2	_	8.99																																							
	26.1		67.0	67.4	67.8	68.2	9.89	0.69	69.3	69.7	70.1	70.5	70.9	71.3	71.6	72.0	72.4	72.8	73.2	73.6	73-9	74.3	74.7	75.1	75.5	75.9	76.2	20.6	77.0	77.4	77.8	78.2	78.5	78.9	79.3	79.7	80.1	80.5	80.8	81.2	§1.6	
		Sug.	17.5	17.6	17.7	17.8	17.9	18.0	18.1	18.2	18.3	18.4	18.5	18.6	18.7	8.8	18.9	0.61	1.61	19.2	19.3	19.4	19.5	9.61	19.7	8.61	6.61	20.0	20.1	20.2	20.3	20.4	20.5	20.6	20.7	20.8	20.9	21.0	21.1	21.2	21.3	
		P.R.	68.0	68.4	8.8	69.2	9.69	70.0	70.4	70.8	71.2	71.5	71.9	72.3	72.7	73.1	73.5	73.9	74.3	74.7	75.0	75.4	75.8	76.2	26.6	77.0	77.4	77.8	78.2	78.0	29.0	79.3	79.7	80.1	80.5	80.0	81.3	81.7	82.0	82.4	87.8	

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	83.6 84.0	84.0 84.3	84.3 84.6	84.6 85.0	22.5 22.6	88.1	83.0 33.0		83.3		83.3 83.7	83.6 83.3 84.0 83.7	83.9 83.6 83.3 84.3 84.0 83.7
w w	8 3.3 3.3 3.3	83.2 83.2	88 33.58 50.58	88 83.8 8.4.4	22.3 22.4	86.9 87.3	81.9 82.3		82.7 82.6		82.5 82.9	82.8 83.2 82.9	83.1 82.8 82.5 83.5 83.2 82.9
	8 2.2 8 2.5	82.5 82.8	82.8 83.1	83.1 83.5	22.1 22.2	86.5 86.5			80 10 4.00 4.00		81.7 82.1	82.1 81.7 '82.4 82.1	82.8 82.1 81.7 82.8 82.4 82.1
	81.8	82.1	82.4	82.7	22.0	85.7	80.8		81.1		81.4	81.7 81.4	82.0 81.7 81.4

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	2.3		48
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	5.0	2.4.2.4.2.5.6.2.5.6.2.5.6.6.5.6.6.5.6.6.5.6.6.6.6	44 44 44 44 44 44 44 44 44 44 44 44 44
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	Pol.	ױ שׁ שִׁשְׁ שִׁי שִׁ	

TABLE VI.

Amount of Calcium Oxide in Milk of Lime of various densities at 15°C, prepared by Blatner.

Degree Brix.	Degree Baumé.	Weight of one liter Milk of Lime in Grams.	CaO per liter in Grams.	Per Cent CaO.	Degree Brix.	Degree Baumé.	Weight of one liter Milk of Lime in Grams.	CaO per liter in Grams.	Per Cent CaO.
1.8	I	1007	7.5	0.74	29.0	16	1125	159	14.13
3.6	2	1014	16.5	1.64	30.8	17	1134	170	15.00
5.4	3	1022	26	2.54	32.7	18	1142	181	15.85
7.2	4	1029	36	3.50	34.6	19	1152	193	16.75
9.0	5	1037	46	4.43	36.4	20	1162	206	17.72
10.8	6	1045	56	5.36	38.3	21	1171	218	18.61
12.6	7	1052	65	6.18	40.1	22	1180	229	19.40
14.4	8	1060	75	7.08	42.0	23	1190	242	20.34
16.2	9	1067	84	7.87	43.9	24	1200	255	21.25
18.0	10	1075	94	8.74	45.8	25	1210	268	22.15
19.8	11	1083	104	9.60	47.7	26	1220	281	23.03
21.7	12	1091	115	10.54	49.6	27	1231	295	23.96
23.5	13	1100	126	11.45	51.5	28	1241	309	24.90
25.3	14	1108	137	12.35	53.5	29	1252	324	25.87
27.2	15	1116	148	13.26	55.4	30	1263	339	26.84



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